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Electronic Supplementary Information

Solvent-free synthesis of symmetric methylene diesters via direct reaction of armatic carboxylates with 1,*n*-dihaloalkanes

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1. Experimental installation

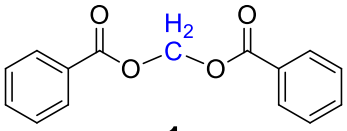
The 80mL Pyrex Vessel is microwave synthesis system accessory of CEM Discover(**Figure 1**), an internal volume of 80mL with a working volume of 50mL. It has a maximum operating temperature of 220°C and a maximum operating pressure of 200 psi (13.789 bar).

Figure 1 80mL Vessel kit

2. Characterization data of the products

Compounds **4, 9, 18, 26** and **29** were known and the data for these compounds have been published in a previous paper we reported.[1] The yield is that of dibromoalkane as reactant. Specific detailed data for each of the other compounds are given below.

Methylene dibenzoate (1)



1

White crystal, (1.192g, yield-93%), recrystallized with ethanol. m.p. 97-98 °C.

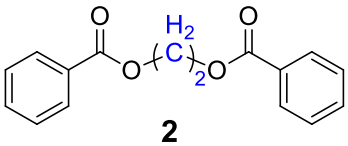
^{1}H NMR (400 MHz, acetone-d_6) δ (ppm): 8.05-8.06 (m, 4H), 7.68 (m, 2H), 7.52-7.67 (m, 4H), 6.26 (s, 2H).

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm): 205.97, 164.79, 133.81, 129.66, 129.08, 128.72, 80.54.

HRMS calcd for $C_{15}H_{12}O_4Na$ $[M + Na]^+$, 279.0634, found 279.0626.

The melting point and ^{1}H NMR data are in good agreement with literature report.[2]

Ethylene dibenzoate(ethanediol dibenzoate) (2)



2

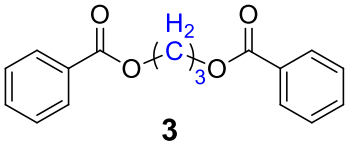
White crystal, (1.189g, yield-88%), recrystallized with ethanol. m.p. 70-71°C.

^{1}H NMR (400 MHz, acetone-d_6) δ (ppm): 8.04-8.05 (m, 4H), 7.62-7.64 (m, 2H), 7.49-7.52 (m, 4H), 4.42 (s, 4H).

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm): 205.94, 165.86, 133.17, 130.04, 129.37, 128.53, 62.74.

HRMS calcd for $C_{16}H_{14}O_4Na$ $[M + Na]^+$, 293.0793, found 293.0782.

1,3-Propanediol-1,3-dibenzoate (trimethylene dibenzoate) (3)



3

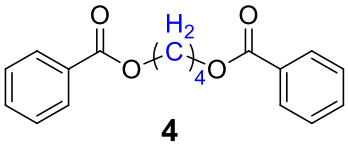
White solid, (1.265g, yield-89%), recrystallized with ethanol. m.p. 56-57 ^{o}C**.**

^{1}H NMR (400 MHz, acetone-d_6) δ (ppm): 8.01-8.02 (m, 4H), 7.59-7.62 (m, 2H), 7.45-7.48(m, 4H), 4.51(t, 4H), 2.25-2.29(m, 2H).

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm):205.95, 165.95, 133.00, 130.27, 129.30, 128.47, 61.80.

HRMS calcd for $C_{17}H_{16}O_4Na$ $[M + Na]^+$, 307.0947, found 307.0938.

1,4-Butamethylene dibenzoate (4)



4

White crystal, (1.268g, yield-85%), recrystallized with ethanol. m.p. 82-83^{o}C

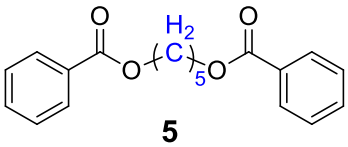
^{1}H NMR (400 MHz, acetone-d_6) δ (ppm): 8.04-8.05 (m, 4H), 7.62-7.64 (m, 2H), 7.49-7.52(m, 4H), 4.41-4.43(t, 4H), 1.96-1.99(m, 4H).

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm): 206.29,166.80, 132.79, 129.47, 129.24, 128.49, 128.39, 64.54, 25.33.

HRMS calcd for $C_{18}H_{18}O_4Na$ $[M + Na]^+$, 321.1103, found 321.1093.

These data are in good agreement with literature report.[1]

1,5-Pentamethylene dibenzoate(1,5-pentanediol-1,5-dibenzoate) (5)



5

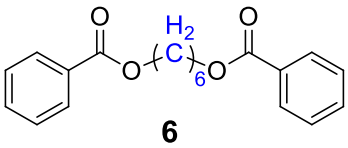
White crystal, (1.405g, yield-90%), recrystallized with ethanol. m.p. 110-112 ^{o}C.

^{1}H NMR (400 MHz, acetone-d_6) δ (ppm): 7.99-8.03(m, 2H), 7.74-7.76(m, 3H), 7.58-7.60(m, 2H), 7.43-7.49(m, 3H), 4.54(m, 4H), 1.73-1.79(m, 2H), 1.36-1.42(m, 2H), 0.92-0.94(m, 2H).

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm): 205.90, 166.39, 132.79, 130.73, 129.47, 129.20, 128.49, 64.25, 25.36.

HRMS calcd for $C_{19}H_{20}O_4Na$ $[M + Na]^+$, 335.1260, found 335.1252.

1,6-Hexanediol dibenzoate (1,6-bis-benzoyloxy-hexane) (6)



6

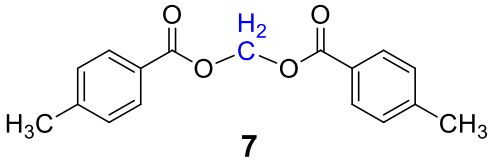
White crystal, (1.501g, yield-92%), recrystallized with ethanol. m.p. 59-60 °C.

^{1}H NMR (400 MHz, acetone-d_6) δ (ppm): 8.00-8.01(m, 4H), 7.60-7.62(m, 2H), 7.47-7.50(m, 4H), 4.30-4.33(m, 4H), 1.79-1.81(m, 4H), 1.54-1.56(m, 4H).

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm): 205.90, 166.00, 132.91, 130.50, 129.21, 128.48, 64.53, 25.54.

HRMS calcd for $C_{20}H_{22}O_4Na$ $[M + Na]^+$, 349.1416, found 349.1407.

Methylene di-*p*-methylbenzoate (7)



7

White crystal, (1.364g, yield-96%), recrystallized with ethanol. m.p. 95-96°C.

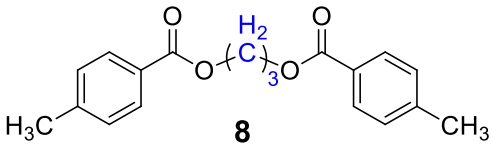
^{1}H NMR (400 MHz, acetone-d_6) δ (ppm): 7.94-7.96 (m, 4H), 7.34-7.35 (m, 4H), 6.24 (s, 2H), 2.40(s, 6H).

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm): 205.89, 164.89, 144.72, 129.72, 129.32, 126.36, 80.15, 20.46.

HRMS calcd for $C_{17}H_{16}O_4Na$ $[M + Na]^+$, 307.0947, found 307.0938.

The melting point and ^{1}H NMR data are in good agreement with literature report.[2]

1,3-Propanediol-di-p-methylbenzoate (8)



8

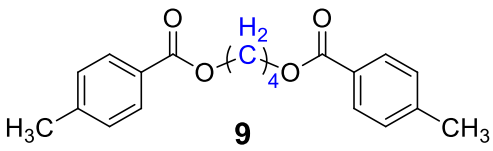
White crystal, (1.437g, yield-92%), recrystallized with ethanol. m.p. 89-90°C.

^{1}H NMR (400 MHz, acetone-d_6) δ (ppm): 7.88-7.90 (m, 4H), 7.26-7.27 (m, 4H), 4.46-4.48(m, 4H), 2.37(s, 6H), 2.22-2.26(m, 2H).

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm): 205.89, 165.97, 143.60, 129.35, 129.06, 127.55, 61.52, 20.58.

HRMS calcd for $C_{19}H_{20}O_4Na$ $[M + Na]^+$, 335.1260, found 335.1250.

1,4-Butamethylene-di-*p*-methylbenzoate (1,4-Butylene glycol di-*p*-methylbenzoate) (9)



9

White crystal, (1.550g, yield-95%), recrystallized with ethanol. m.p. 115-116 °C.

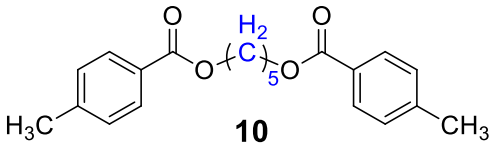
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.93-7.95 (d, J = 8.0 Hz, 4H), 7.23-7.25 (d, J = 8.0 Hz, 4H), 4.37-4.41 (t, J = 5.6 Hz, 4H), 2.42 (s, 6H),1.93-1.96 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 166.6, 143.6, 129.6, 129.1, 127.6, 64.3, 25.6, 21.6.

MS (*m/z*): Calculated for $C_{20}H_{22}O_4$: 326.15, Found: 326.

These data have been reported in the literature 1.

1,5-Pentamethylene-di-p-methylbenzoate (10)



10

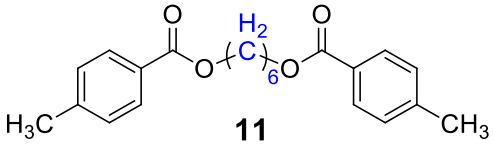
White crystal, (1.531g, yield-90%), recrystallized with ethanol. m.p. 49-51 ºC.

^{1}H NMR (400 MHz, acetone-d_6) δ (ppm): 7.87-7.89 (m, 4H), 7.25-7.27 (m, 2H), 4.30(m, 4H), 2.37(s, 6H), 1.80-1.85 (m, 4H), 1.59-1.64 (m, 2H).

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm):205.86, 165.86, 143.46, 129.29, 129.08, 127.77, 64.55, 22.42, 20.68.

HRMS calcd for $C_{21}H_{24}O_4Na$ $[M + Na]^+$, 363.1573, found 363.1562.

1,6-Hexanediol-di-p-methylbenzoate(11)



11

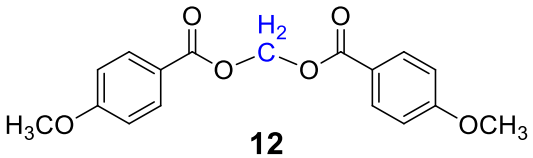
White crystal, (1.630g, yield-92%), recrystallized with ethanol. m.p. 84.5-85.5 ºC.

^{1}H NMR (400 MHz, acetone-d_6) δ (ppm): 7.88-7.89 (m, 4H), 7.27-7.29 (m, 4H), 4.27-4.29(m, 4H), 2.37(s, 6H), 1.77-1.79 (m, 4H), 1.53-1.54 (m, 4H).

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm): 205.93, 165.94, 143.52, 129.19, 128.99, 127.75, 64.07, 25.54, 20.65.

HRMS calcd for $C_{22}H_{26}O_4Na$ $[M + Na]^+$, 377.1729, found 377.1718.

Methylene di-*p*-methoxylbenzoate (12)



12

White crystal, (1.518g, yield-96%), recrystallized with ethanol. m.p. 112-113ºC.

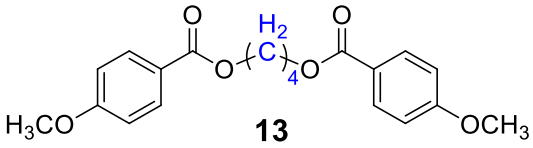
^{1}H NMR (400 MHz, acetone-d_6) δ (ppm): 7.99-8.00 (m, 4H), 7.03-7.04 (m, 4H), 6.18 (s, 2H), 3.87(s, 6H).

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm): 206.14, 164.15, 131.78, 121.21, 114.05, 113.45, 80.14, 55.07.

HRMS calcd for $C_{17}H_{16}O_6Na$ $[M + Na]^+$, 339.0845, found 339.0838.

The melting point and ^{1}H NMR data are in good agreement with literature report.[2]

1,4-Butamethylene-di-*p*-methoxylbenzoate (13)



13

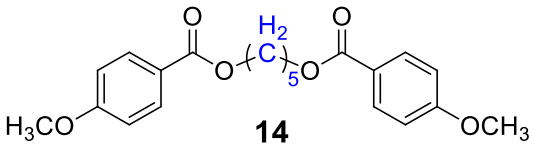
White crystal, (1.756g, yield-98%), recrystallized with ethanol. m.p. 150-151°C.

^{1}H NMR (400 MHz, acetone-d_6) δ (ppm): 7.96-7.98 (m, 4H), 7.00-7.02 (m, 4H), 4.34-4.37 (t, 4H), 3.87 (s, 6H), 1.92-1.94 (m, 4H).

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm): 165.71, 163.63, 131.39, 122.76, 113.78, 64.02, 55.09, 25.50.

HRMS calcd for $C_{20}H_{22}O_6Na$ $[M + Na]^+$, 381.1314, found 381.1309.

1,5-Pentamethylene -di-*p*-methoxylbenzoate (14)



14

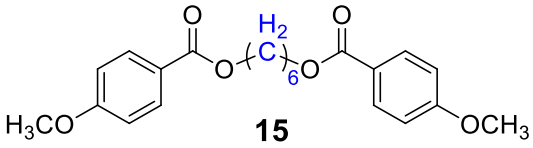
White crystal, (1.806g, yield-97%), recrystallized with ethanol. m.p. 70-71°C.

^{1}H NMR (400 MHz, acetone-d_6) δ (ppm): 7.94-7.96 (m, 4H), 6.98-7.00 (m, 4H), 4.29-4.32 (t, 4H), 3.86 (s, 6H), 1.80-1.86 (m, 4H), 1.61-1.65 (m, 2H).

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm): 165.77, 163.58, 131.36, 122.81, 113.78, 64.15, 55.10, 28.29, 22.50.

HRMS calcd for $C_{22}H_{27}O_6$ $[M + H]^+$, 387.1808, found 387.1807.

1,6-Hexanediol-di-*p*-methoxylbenzoate (15)



15

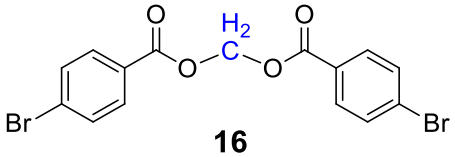
White crystal, (1.855g, yield-96%), recrystallized with ethanol. m.p. 89-90 °C.

^{1}H NMR (400 MHz, acetone-d_6) δ (ppm): 7.95-7.97 (m, 4H), 6.99-7.01 (m, 4H), 4.26-4.29 (t, 4H), 3.86 (s, 6H), 1.77-1.80 (m, 4H), 1.53-1.56 (m, 4H).

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm): 165.76, 163.58, 131.35, 122.85, 113.78, 64.31, 55.09, 28.60, 25.67.

HRMS calcd for $C_{21}H_{24}O_6Na$ $[M + Na]^+$, 395.1471, found 395.1465.

Methylene di-*p*-bromobenzoate (16)



16

White solid. (1.966g, yield-95%), recrystallized with ethanol. m.p. 118-119 °C.

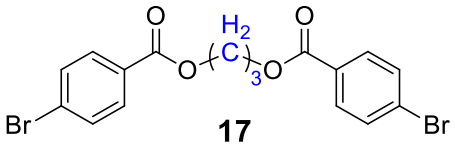
^{1}H NMR (400 MHz, acetone-d_6) δ (ppm): 7.95-7.98 (m, 4H), 7.71-7.74 (m, 4H), 6.25 (s, 2H).

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm): 205.83, 164.19, 132.12, 131.58, 128.28, 80.58.

HRMS calcd for $C_{15}H_{10}Br_2O_4Na$ $[M + Na]^+$ 434.88381, found 434.88382.

These data are in good agreement with literature report.[3]

1,3-Propanediol-di-*p*-bromobenzoate(17)



17

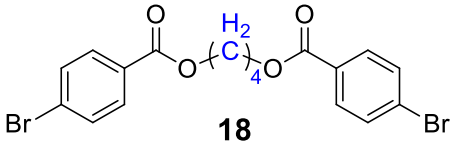
White solid. (2.012g, yield-91%), recrystallized with ethanol. m.p. 102-103 °C.

^{1}H NMR (400 MHz, acetone-d_6) δ (ppm): 7.90-7.93 (m, 4H), 7.65-7.68 (m, 4H), 4.49-4.52(t, 4H), 2.24-2.31 (m, 2H).

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm): 205.86, 165.26, 131.82, 131.23, 129.48, 127.51, 62.22.

HRMS calcd for $C_{17}H_{14}Br_2O_4K$ $[M + K]^+$, 478.88904, found 478.88874.

1,4-Butamethylene di-p-bromobenzoate (18)



18

White crystal, (2.016g, yield-88%), recrystallized with ethanol. m.p. 127-128 °C.

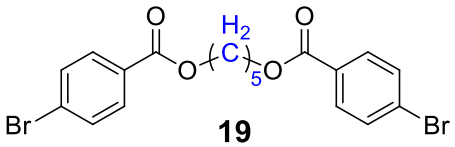
^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.87-7.89 (m, 4H). 7.55-7.58 (m, 4H), 4.39 (m, 4H), 1.92-1.94 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 165.9, 131.2, 131.8, 129.2, 128.2, 64.8, 25.6.

MS (m/z): Calculated for $C_{18}H_{16}Br_2O_4$: 455.94, Found: 456.

These data have been reported in the literature 1.

1,5-Pentamethylene-di-*p*-bromobenzoate (19)



19

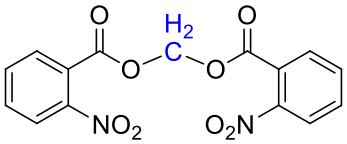
White crystal, (1.998g, yield-85%), recrystallized with ethanol. m.p. 45-46 °C.

^{1}H NMR (400 MHz, acetone-d_6) δ (ppm): 7.90-7.94 (m, 4H), 7.66-7.69 (m, 4H), 4.33-4.37(t, 4H), 1.82-1.90 (m, 4H), 1.62-1.66 (m, 2H).

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm): 205.62, 165.26, 131.85, 131.17, 129.71, 127.40, 64.78, 22.38.

HRMS calcd for $C_{19}H_{19}Br_2O_4$ $[M + H]^+$ 468.96446, found 468.96431.

Methylene di-*o*-nitrobenzoate (20)



20

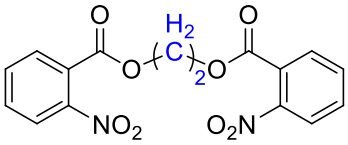
Light yellow crystal, (1.574g, yield-91%), recrystallized with ethanol. m.p. 90.5-91.5 °C.

^{1}H NMR (400 MHz, acetone-d_6) δ (ppm): 7.94-8.10 (m, 4H), 7.88-7.92 (m, 4H), 6.21 (s, 2H).

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm): 205.35, 163.73, 133.58, 133.20, 130.29, 125.98, 124.30, 80.62.

HRMS calcd for $C_{15}H_{10}N_2O_8Na$ $[M + Na]^+$ 369.03294, found 369.03293.

Ethanediol di-*o*-nitrobenzoate(21)



21

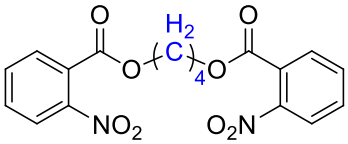
Light yellow crystal, (1.675g, yield-93%), recrystallized with ethanol. m.p. 135.5-136.5 °C.

^{1}H NMR (400 MHz, acetone-d_6) δ (ppm): 7.91-8.02 (m, 4H), 7.83-7.87 (m, 4H), 4.67 (s, 4H).

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm): 205.94, 164.72, 133.30, 132.78, 130.21, 126.63, 124.04, 63.68.

HRMS calcd for $C_{16}H_{12}N_2O_8K$ $[M + K]^+$ 399.02252, found 399.02251.

1,4-Butamethylene di-*o*-nitrobenzoate(22)



22

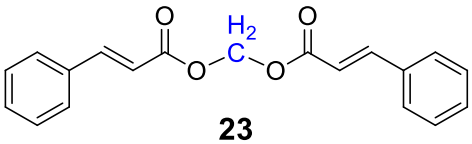
Light yellow crystal, (1.825g, yield-94%), recrystallized with ethanol. m.p. 86-87 °C.

^{1}H NMR (400 MHz, acetone-d_6) δ (ppm): 7.88-8.01 (m, 4H), 7.80-7.87 (m, 4H), 4.37-4.40(m, 4H), 1.86-1.89 (m, 4H).

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm): 206.12, 164.90, 133.27, 132.58, 130.07, 127.13, 123.98, 65.62, 24.90.

HRMS calcd for $C_{18}H_{16}N_2O_8K$ $[M + K]^+$ 427.05382, found 427.05384.

Methylene di-cinnamate (23)



23

White crystal, (1.403g, yield-91%), recrystallized with ethanol. m.p. 83-84°C.

IR (KBr) *v*: 3020, 2955, 2868, 1930, 1713, 1610, 1473, 1443, 1271, 1018, 965, 754, 690 cm^{-1}.

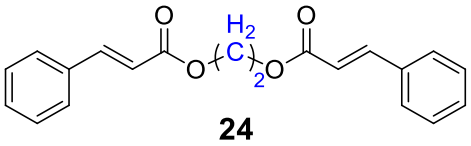
^{1}H NMR (400 MHz, acetone-d_6) δ (ppm): 7.77-7.79 (d, 2H), 7.69-7.70 (m, 4H), 7.43-7.44(m, 4H), 6.57-6.59 (d, 2H), 6.01(s, 2H).

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm): 205.99, 165.05, 146.43, 134.15, 130.75, 128.92, 128.38, 116.83, 79.36.

HRMS calcd for $C_{19}H_{16}NaO_4$ $[M + Na]^+$, 331.0946, found 331.0939.

The melting point and ^{1}H NMR data are in good agreement with literature report.[2]

Ethanediol di-cinnamate (24)



24

White crystal, (1.434g, yield-89%), recrystallized with ethanol. m.p. 90-91ºC.

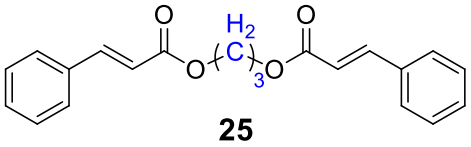
IR (KBr) *v*: 3037, 2955, 2868, 1930, 1713, 1610, 1461, 1443, 1271, 1035, 965, 754, 691 cm^{-1}.

^{1}H NMR (400 MHz, acetone-d_6) δ (ppm): 7.70-7.73 (d, 2H), 7.66-7.67 (m, 4H), 7.41-7.42(m, 6H), 6.56-6.58 (d, 2H), 4.48(s, 4H).

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm): 205.97, 166.22, 145.02, 134.29, 130.46, 128.93, 128.16, 117.70, 62.22.

HRMS calcd for $C_{20}H_{18}NaO_4$ $[M + Na]^+$, 345.1103, found 345.1094.

1,3-Propanediol-di-cinnamate (25)



25

White crystal, (1.463g, yield-87%), recrystallized with ethanol. m.p. 89-90ºC.

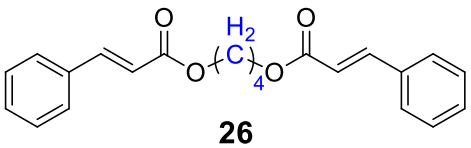
IR (KBr) *v*: 3032, 2955, 2868, 1930, 1712, 1610, 1468, 1440, 1271, 1024, 965, 754, 690 cm^{-1}.

^{1}H NMR (400 MHz, acetone-d_6) δ (ppm): 7.65-7.72 (m, 6H), 7.39-7.42 (m, 6H), 6.54-6.58 (d, 2H), 4.32-4.35(t, 4H), 2.09-2.15(m, 2H).

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm): 205.87, 166.30, 144.64, 134.51, 130.39, 128.99, 128.23, 118.11, 61.28.

HRMS calcd for $C_{21}H_{21}O_4$ $[M + H]^+$ 337.14344, found 337.14341.

1,4-Butamethylene di-cinnamate (1,4-Butylene glycol dicinnamate) (26)



26

White crystal, (1.576g, yield-90%), recrystallized with ethanol. m.p. 92-93ºC.

IR (KBr) *v*: 3028, 2961, 2863, 1930, 1710, 1609, 1466, 1457, 1273, 1021, 965, 755, 690 cm^{-1}.

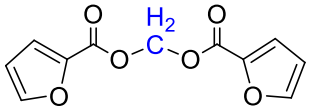
^1H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.66-7.70 (d, *J* = 16.0 Hz, 2H), 7.50-7.53 (m, 4H), 7.36-7.38 (m, 6H), 6.42-6.46 (d, *J* = 16.0 Hz, 2H), 4.25-4.28 (m, 4H), 1.82-1.86 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 167.1, 144.9, 134.5, 130.4, 129.0, 128.2, 118.1, 64.2, 25.6.

MS (*m/z*): Calculated for $C_{22}H_{22}O_4$: 350.15, Found: 350.

These data have been reported in the literature 1.

Metylene di-2-furoate (27)



27

White crystal, (1.026g, yield-87%), recrystallized with ethanol. m.p. 124-125 ^oC.

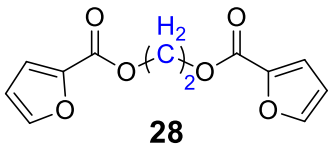
^1H NMR (400 MHz, acetone-d_6) δ (ppm): 7.87-7.88 (s, 2H), 7.36-7.37 (s, 2H), 6.67-6.68 (s, 2H), 6.17 (s, 2H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm): 205.88, 156.58, 148.14, 143.48, 119.74, 112.38, 79.40.

HRMS calcd for $C_{11}H_9O_6$ $[M + H]^+$ 237.03936, found 237.03936.

The melting point, ^1H NMR and ^{13}C NMR data are in good agreement with literature report.[4]

Ethanediol di-2-furoate (28)



28

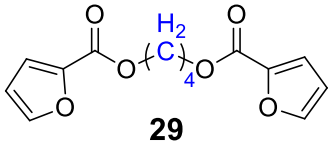
White crystal, (1.050g, yield-84%), recrystallized with ethanol. mp 109-111 ^oC.

^1H NMR (400 MHz, acetone-d_6) δ (ppm): 7.80-7.81 (s, 2H), 7.24-7.25 (s, 2H), 6.62-6.64 (s, 2H), 4.61 (s, 4H) ppm.

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm): 206.11, 157.99, 147.27, 118.39, 112.08, 62.50.

HRMS calcd for $C_{12}H_{11}O_6$ $[M + H]^+$ 251.05501, found 251.05503.

1,4-Butamethylene di-2-furoate (1,4-Butylene glycol di-2-furoate) (29)



29

White crystal, (1.127g, yield-81%), recrystallized with ethanol. m.p. 61-62^oC.

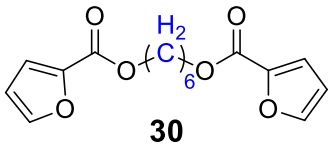
^1H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.55-7.57 (m, 2H), 7.15-7.18 (m, 2H), 6.48-6.51 (m, 2H), 4.30-4.40 (m, 4H), 1.89-1.92 (m, 4H).

^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 158.7, 146.3, 144.7, 117.9, 111.8, 64.4, 25.4.

MS (m/z): Calculated for $C_{14}H_{14}O_6$: 278.08, Found: 278.

These data have been reported in the literature 1.

1,6-Hexanediol-di-2-furoate (30)



30

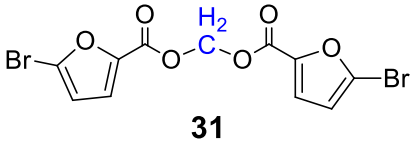
White crystal, (1.195g, yield-78%), recrystallized with ethanol. m.p. 71-72°C.

^{1}H NMR (400 MHz, acetone-d_6) δ (ppm): 7.79 (s, 2H), 7.21-7.22 (s, 2H), 6.61-6.63 (s, 2H), 4.26-4.29(t, 4H), 1.73-1.78(m, 4H), 1.49-1.52 (m, 4H).

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm): 206.11, 158.27, 146.93, 144.87, 117.77, 111.97, 64.45, 25.47.

HRMS calcd for $C_{16}H_{19}O_6$ $[M + H]^+$ 307.11761, found 307.11746.

Ethanediol di-2-(5-bromo)furoate (31)



31

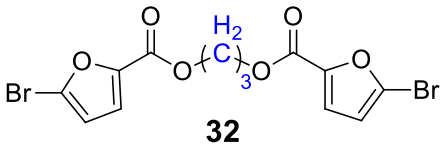
White crystal, (1.832 g, yield-93%), recrystallized with ethanol. m.p. 159-160°C.

^{1}H NMR (400 MHz, acetone-d_6) δ (ppm): 7.38-7.39 (s, 2H), 6.76-6.77 (s, 2H), 6.15 (s, 2H).

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm): 155.51, 145.19, 128.57, 122.11, 114.80, 79.66.

HRMS calcd for $C_{11}H_6Br_2O_6Na$ $[M + Na]^+$ 414.8429, found 414.8425.

1,3-Propanediol-di-2-(5-bromo)furoate (32)



32

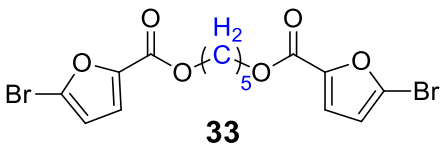
White crystal, (2.004 g, yield-95%), recrystallized with ethanol. m.p. 152-153 °C.

^{1}H NMR (400 MHz, acetone-d_6) δ (ppm): 7.24-7.25 (s, 2H), 6.69-6.70 (s, 2H), 4.43-4.46 (t, 4H), 2.17-2.23 (m, 2H).

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm): 157.03, 146.45, 127.14, 120.33, 114.35, 61.89, 27.94.

HRMS calcd for $C_{13}H_{10}Br_2O_6Na$ $[M + Na]^+$ 446.8701, found 446.8695.

1,5-Pentamethylene-di-(5-bromo)furoate (33)



33

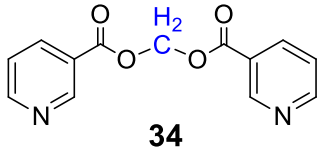
White crystal, (2.160 g, yield-96%), recrystallized with ethanol. m.p. 60-61 °C.

^{1}H NMR (400 MHz, acetone-d_6) δ (ppm): 7.23-7.24 (s, 2H), 6.70-6.71 (s, 2H), 4.29-4.32 (t, 4H), 1.78-1.85 (m, 4H), 1.59 (m, 2H).

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm): 157.11, 146.65, 127.00, 120.09, 114.31, 64.62, 28.12, 22.20.

HRMS calcd for $C_{15}H_{14}Br_2O_6Na$ $[M + Na]^+$ 474.9014, found 474.9008.

Methylene di-3-picolinate (34)



34

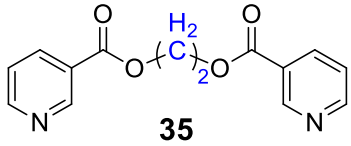
Brown crystal, (0.877g, yield-68%), recrystallized with ethanol. m.p. 79-80 °C.

^{1}H NMR (400 MHz, acetone-d_6) δ (ppm): 9.17-9.18 (d, 2H), 8.82-8.83 (d, 2H), 8.36-8.39 (m, 2H), 7.56-7.59 (m, 2H), 6.32 (s, 2H).

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm): 206.02, 163.90, 154.27, 150.70, 137.28, 123.89, 80.54.

HRMS calcd for $C_{13}H_{11}N_2O_4$ $[M + H]^+$ 259.07133, found 259.07095.

Ethanediol di-3-picolinate (35)



35

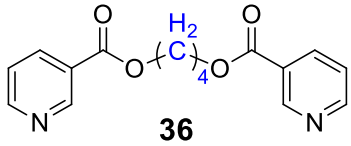
Brown crystal, (0.844g, yield-62%), recrystallized with ethanol. m.p. 127-128 °C.

^{1}H NMR (400 MHz, acetone-d_6): δ (ppm): 9.14-9.15 (d, 2H), 8.78-8.79 (d, 2H), 8.32-8.35 (m, 2H), 7.52-7.55 (m, 2H), 4.76 (s, 4H).

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm): 205.97, 164.93, 153.72, 150.49, 136.94, 123.75, 64.13.

HRMS calcd for $C_{14}H_{13}N_2O_4$ $[M + H]^+$ 273.08698, found 273.08658.

1,4-Butamethylene-di-3-picolinate (36)



36

Brown crystal, (0.901g, yield-60%), recrystallized with ethanol. m.p. 96-97 °C.

^{1}H NMR (400 MHz, acetone-d_6): δ (ppm): 9.14 (d, 2H), 8.78-8.79 (d, 2H), 8.31-8.34 (m, 2H), 7.51-7.54 (m, 2H), 4.44-7.47 (t, 4H), 2.00-2.03 (m, 4H).

^{13}C NMR (100 MHz, acetone-d_6) δ (ppm): 164.95, 153.57, 150.43, 136.73, 126.28, 123.63, 64.78, 25.28.

HRMS calcd for $C_{16}H_{16}N_2O_4$ $[M + H]^+$ 323.1008, found 323.1002.

3. References

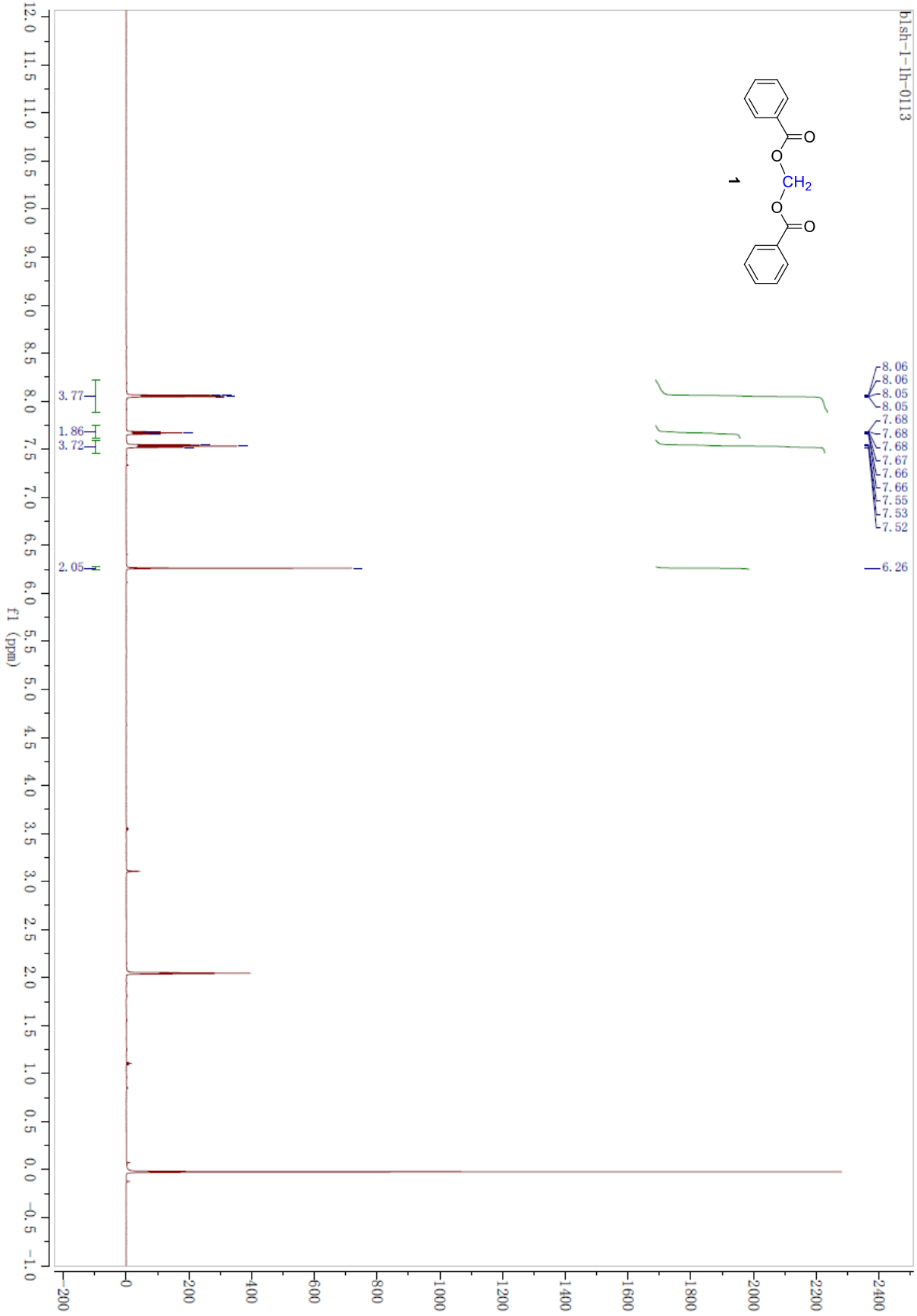
(1) Ding, S.-L.; Bai, L.; Cong, Y.; Cheng, T. *Synth. Commun.* **2018**, *48*, 2559.

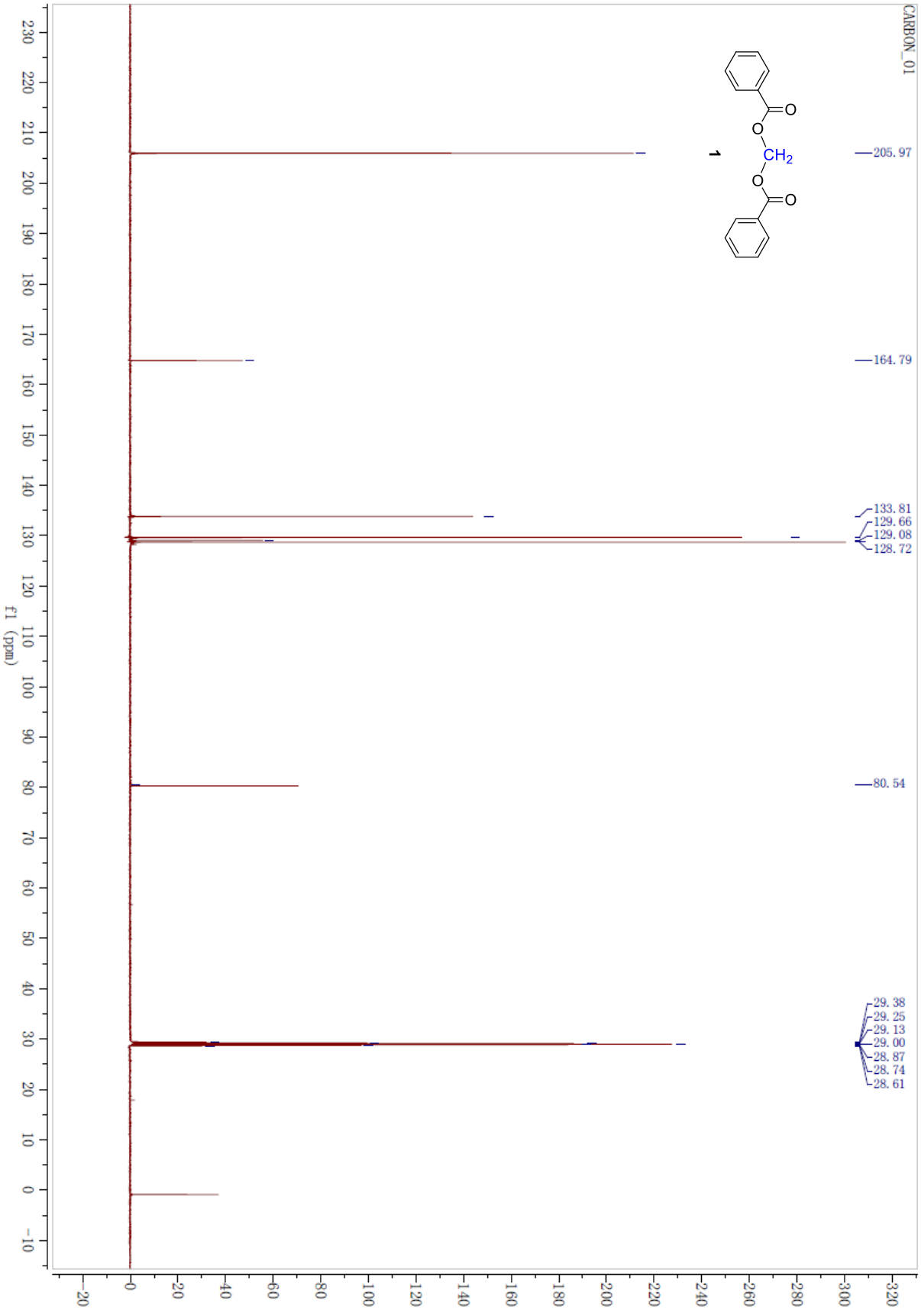
(2) Kavitake, B. P.; Salunkhe, M. M.; Wadgaonkar, P. P. *Synth. Commun.* **1997**, *27*, 1703.

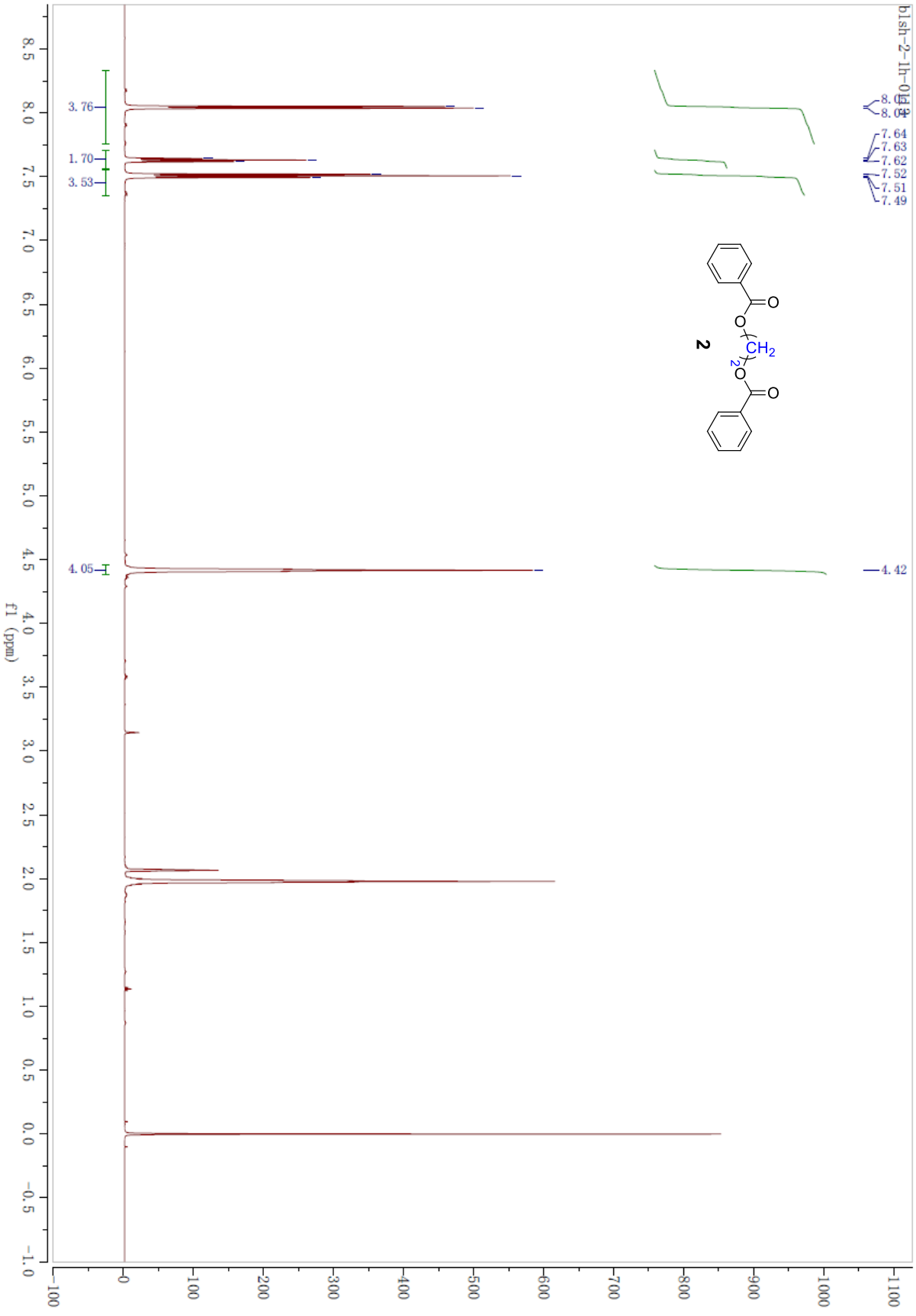
(3) Lin, F.; Feng, Q.; Cui, X. Song, Q. *RSC Adv.* **2013**, *3*, 20246.

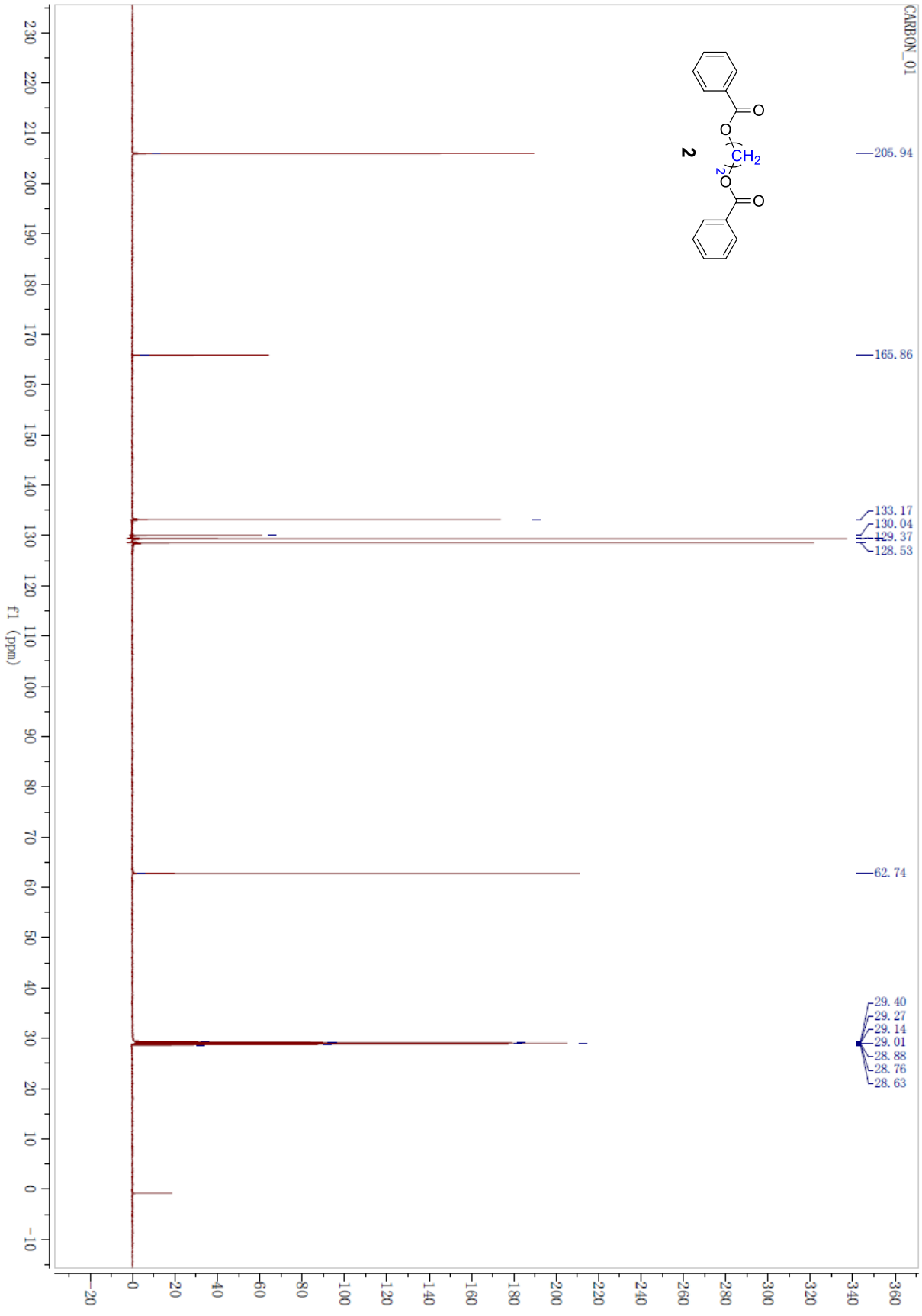
(4) Gómora-Herrera, D.; Lijanova, I. V.; Olivares-Xometl, O.; Toscano, A.; Likhanova, N. V. *Can. J. Chem.* **2017**, *95*, 744.

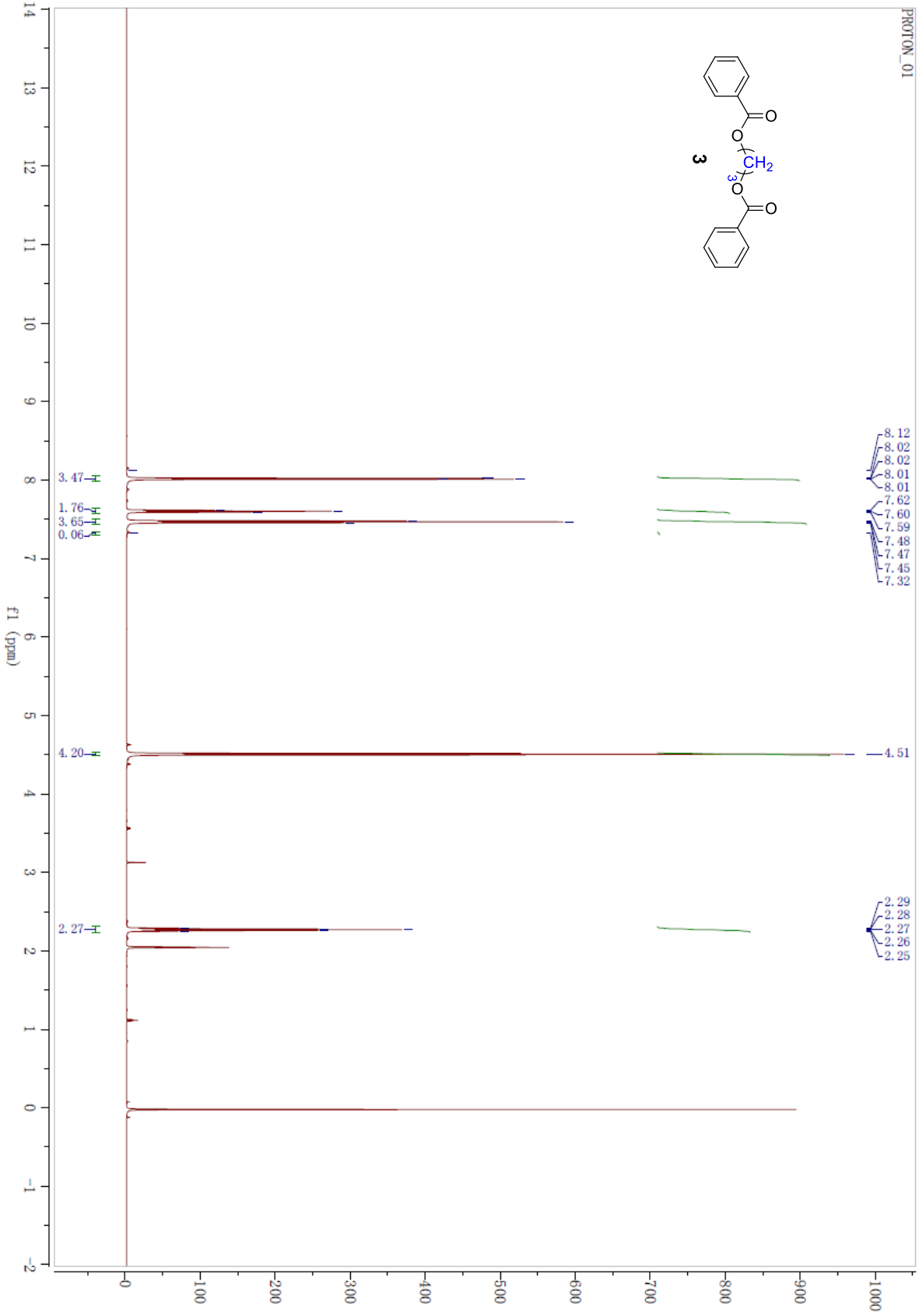
4. ^{1}H and ^{13}C NMR Spectra of the Products

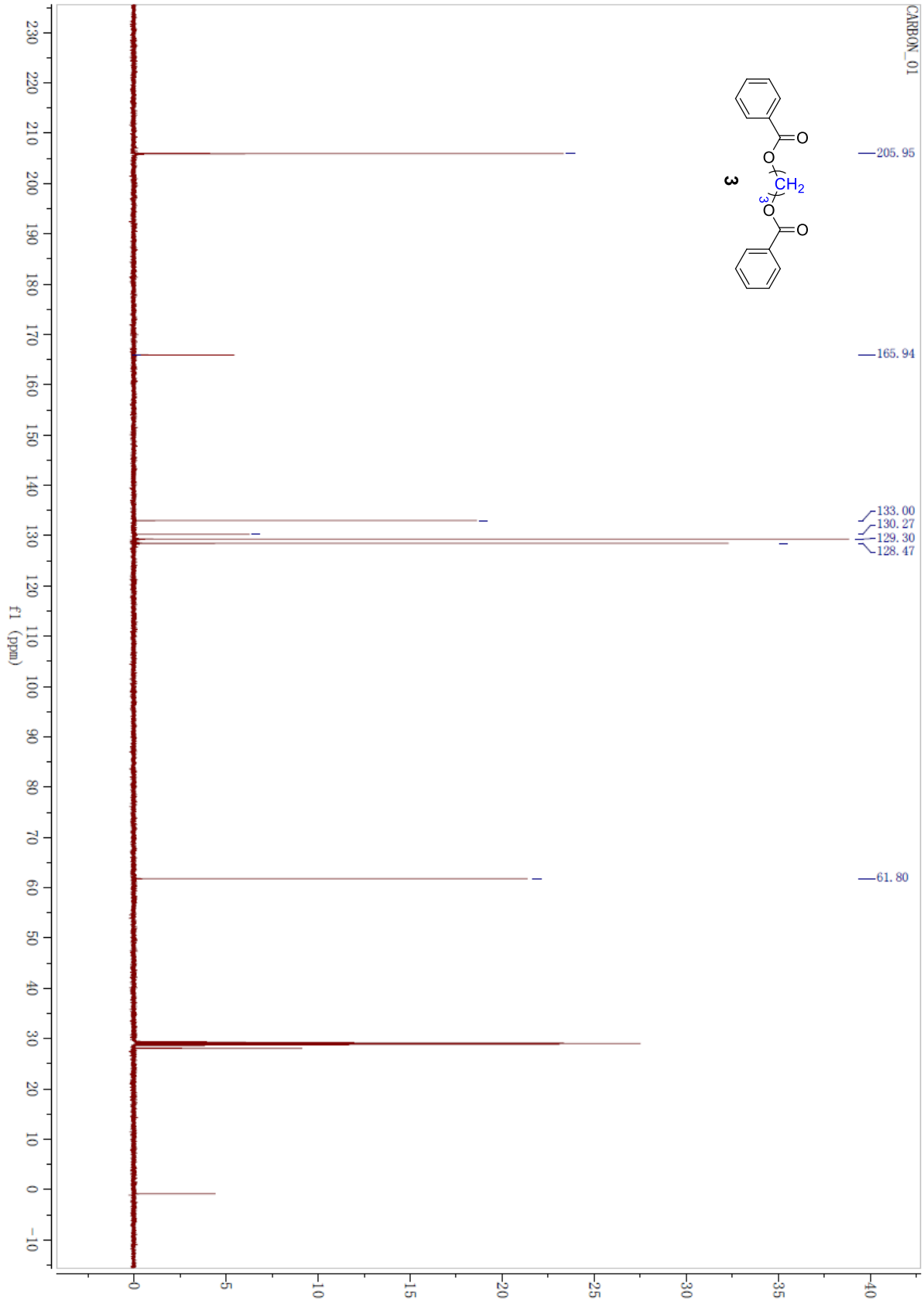


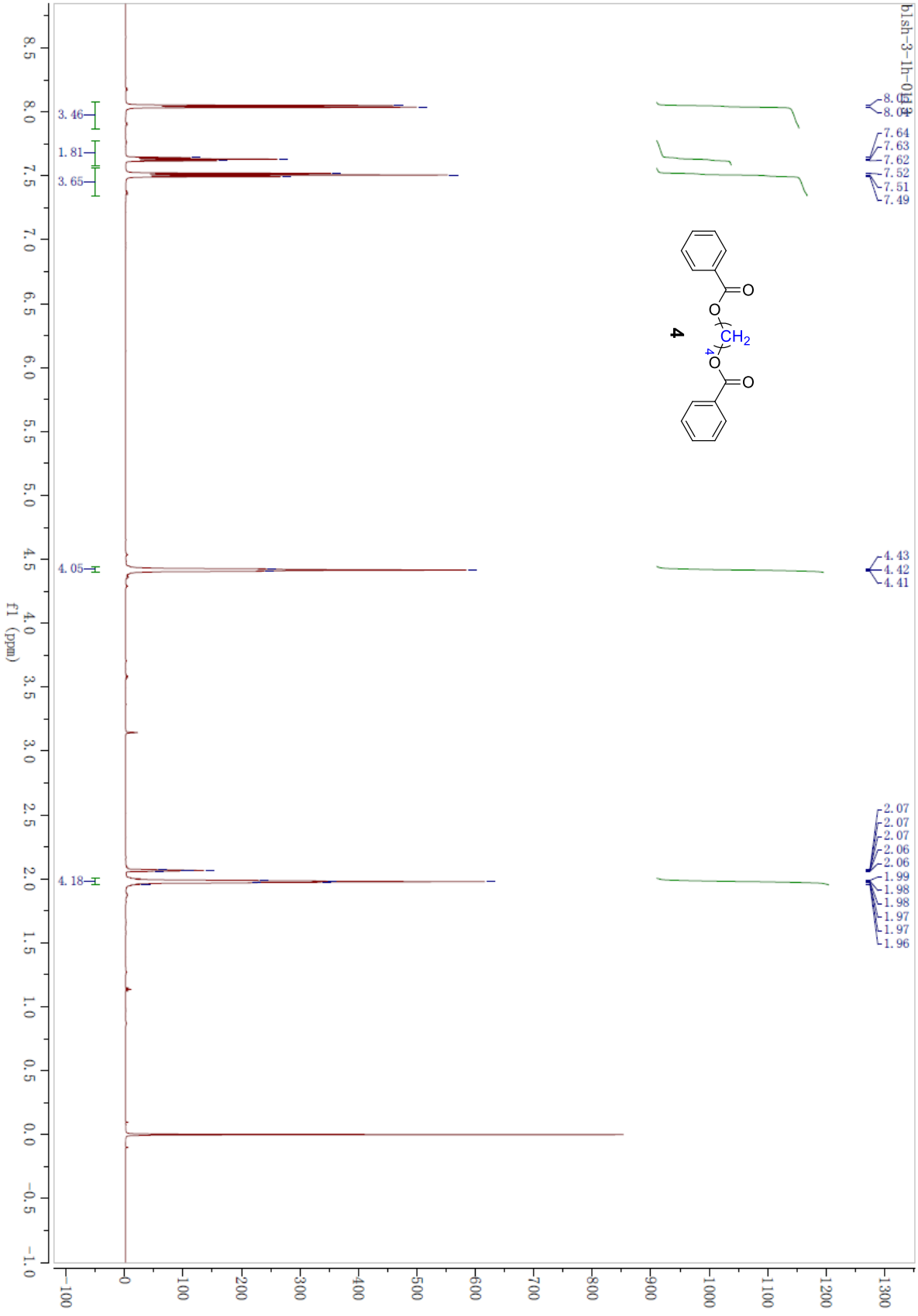

CARBON_01
205.97
164.79
133.81
129.66
129.08
128.72
80.54
29.38
29.25
29.13
29.00
28.87
28.74
28.61
f1 (ppm)

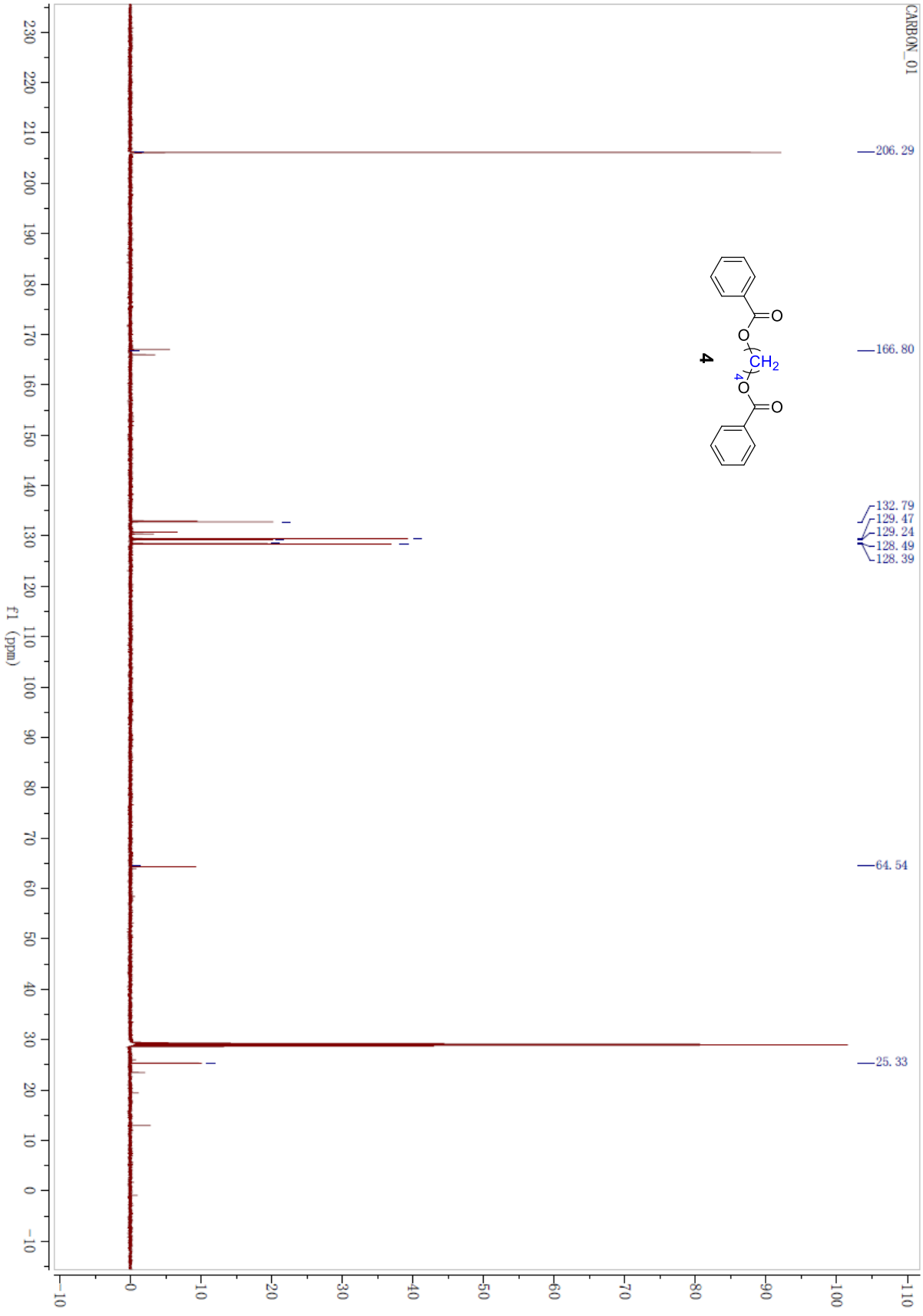

bish-2-1h-0d3
8.04
8.04
7.64
7.63
7.62
7.52
7.51
7.49
4.42
3.76
1.70
3.53
4.05
2
O
O
CH2
2
O
O
f1 (ppm)

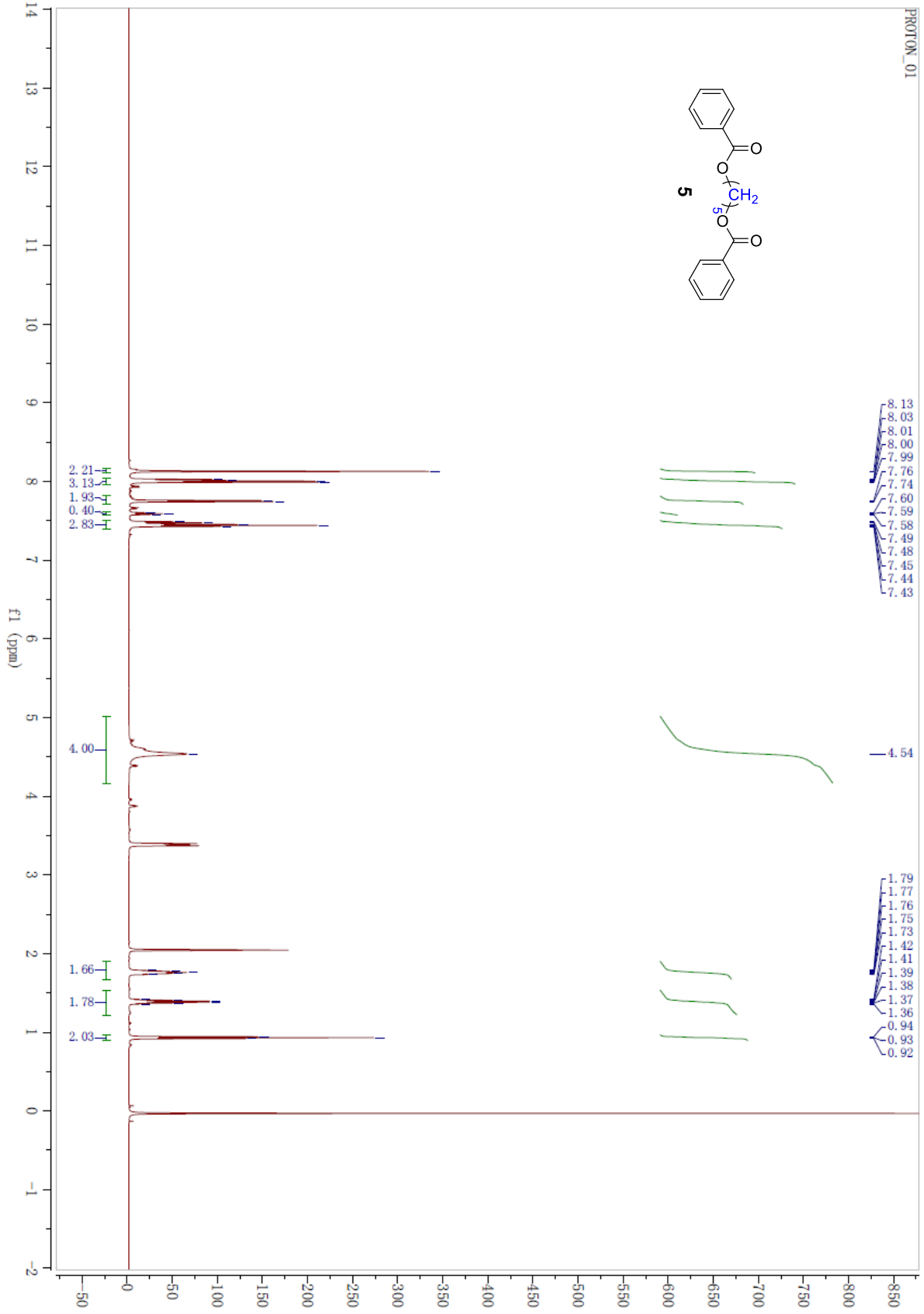

CARBON_01
205.94
165.86
133.17
130.04
129.37
128.53
62.74
29.40
29.27
29.14
29.01
28.88
28.76
28.63
f1 (ppm)

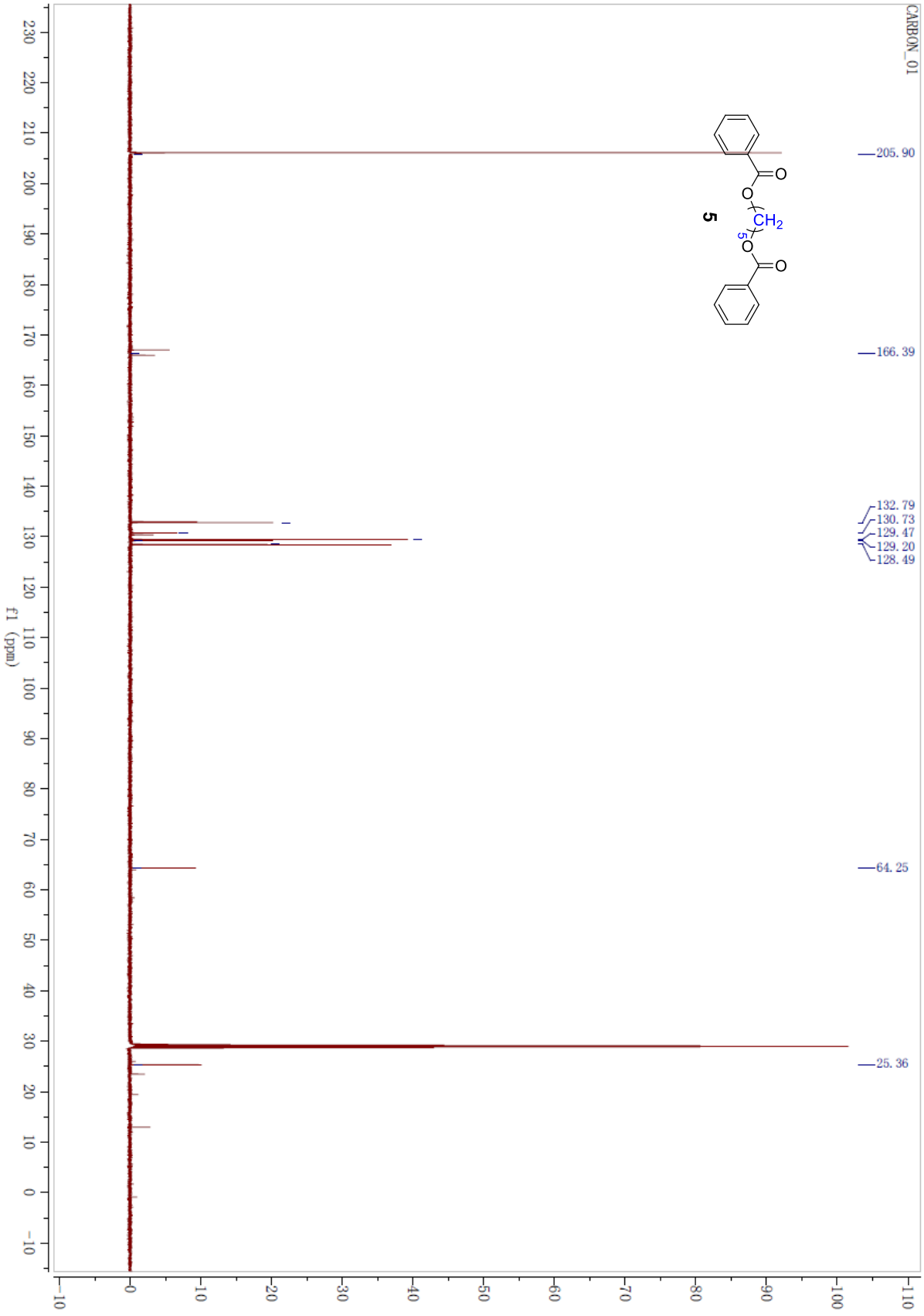

PROTON_01
f1 (ppm)
3.47
1.76
3.65
0.06
4.20
2.27
8.12
8.02
8.02
8.01
8.01
7.62
7.60
7.59
7.48
7.47
7.45
7.32
4.51
2.29
2.28
2.27
2.26
2.25
3
CH2

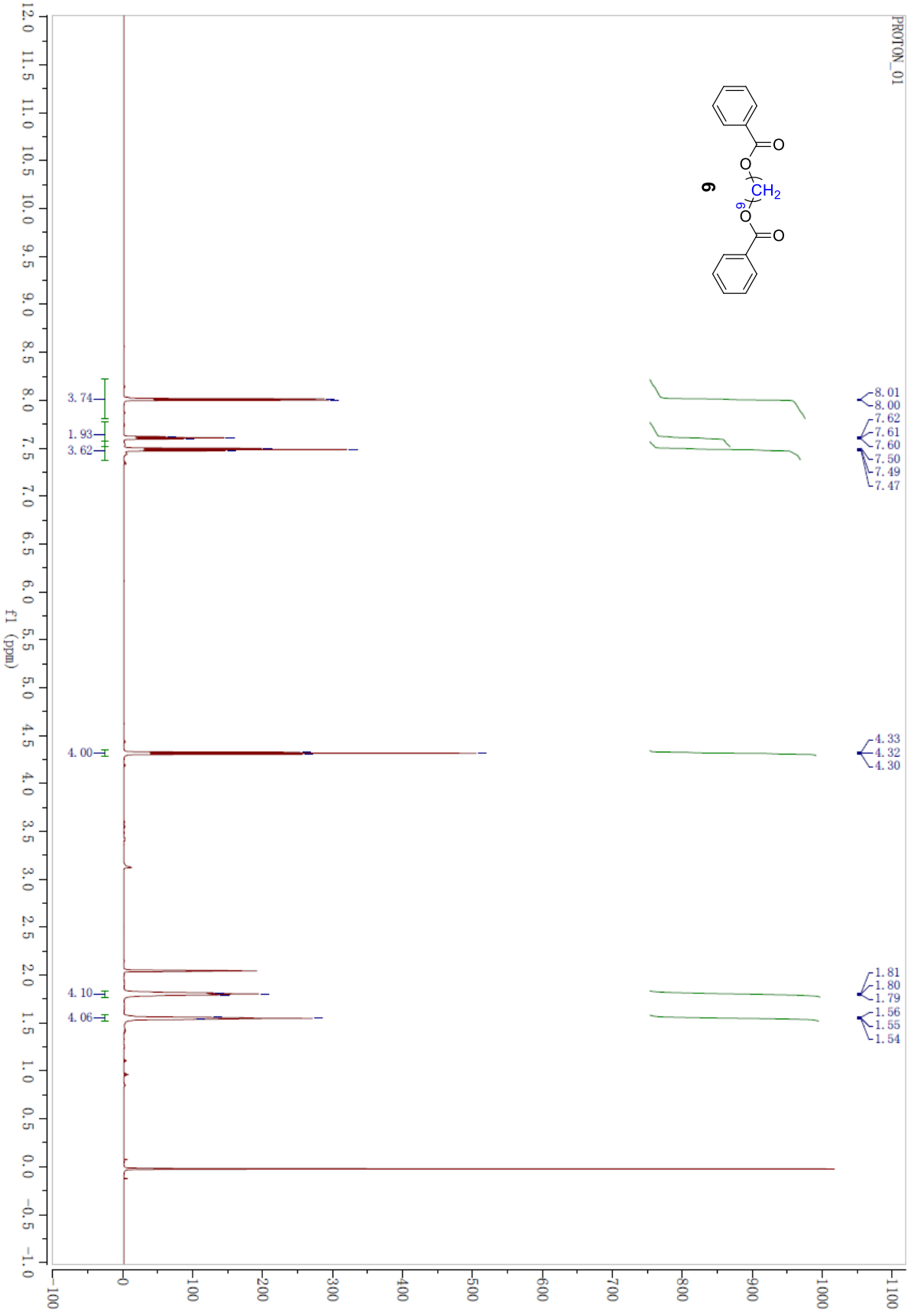

CARBON_01
205.95
165.94
133.00
130.27
129.30
128.47
61.80
f1 (ppm)

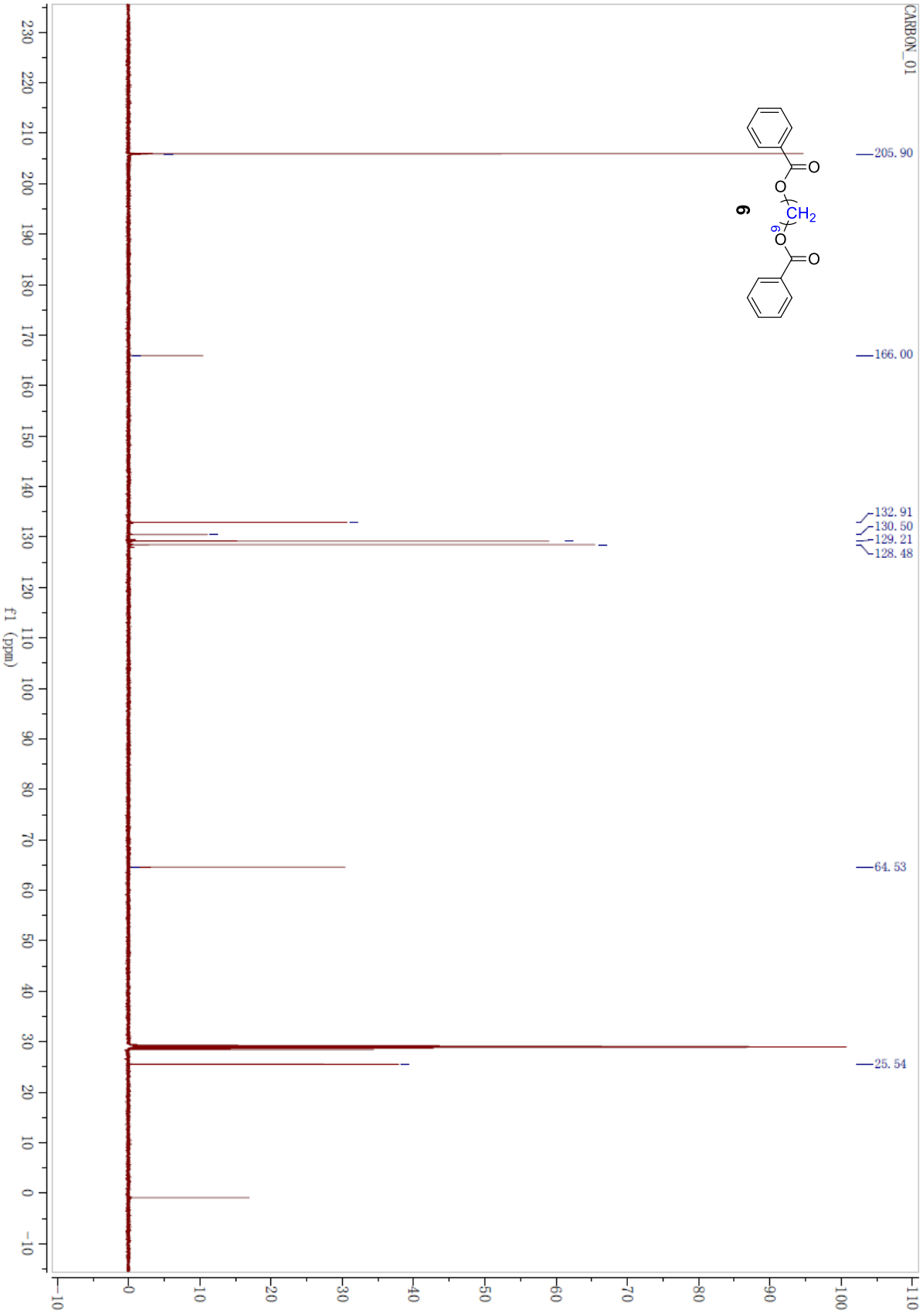

blsh-3-1h-01.1r
8.04
8.03
7.64
7.63
7.62
7.52
7.51
7.49
4.43
4.42
4.41
2.07
2.07
2.07
2.06
2.06
1.99
1.98
1.98
1.97
1.97
1.96
3.46
1.81
3.65
4.05
4.18
f1 (ppm)
O
O
CH2
4
O
O

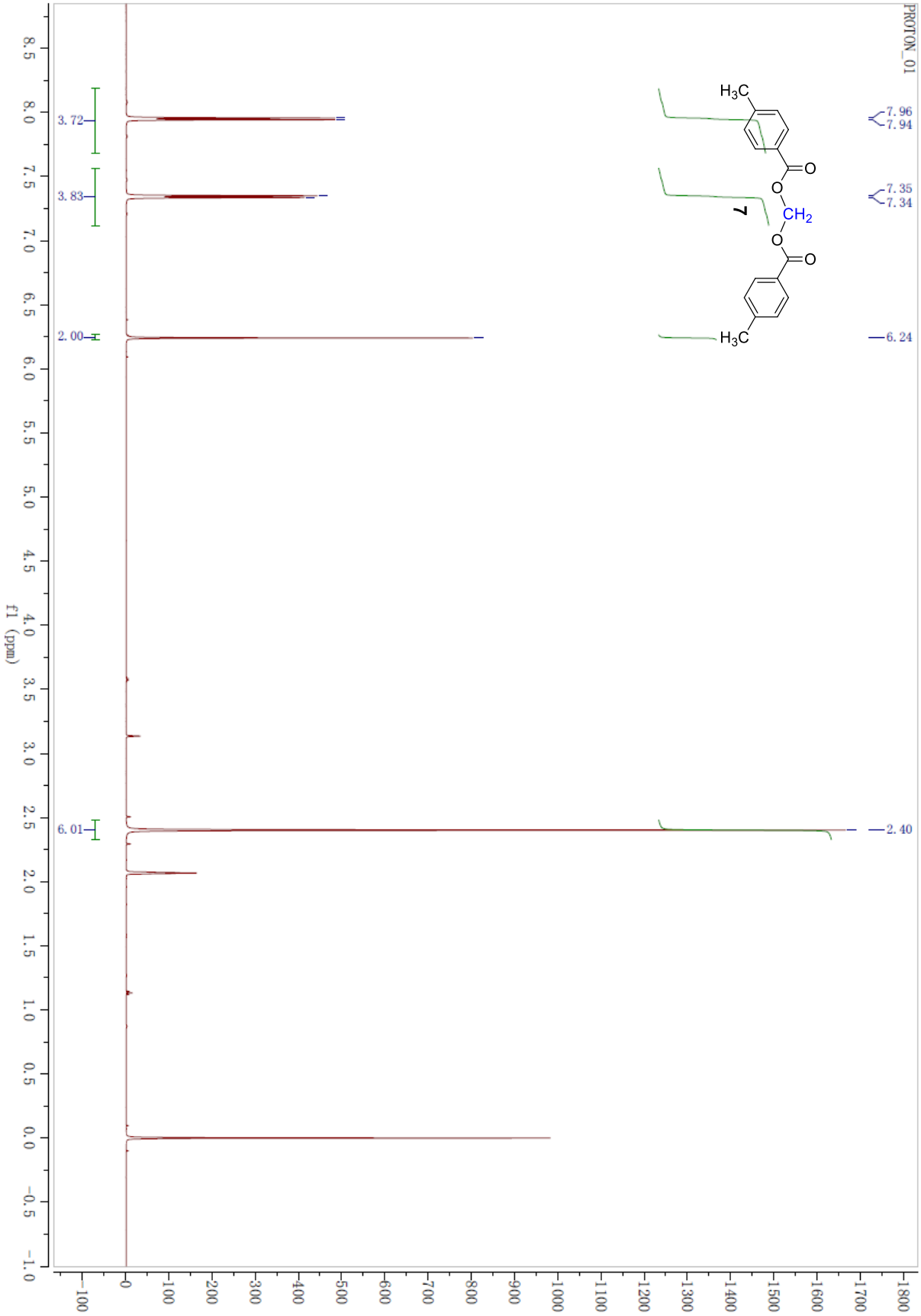

CARBON_01
206.29
166.80
132.79
129.47
129.24
128.49
128.39
64.54
25.33
f1 (ppm)
4

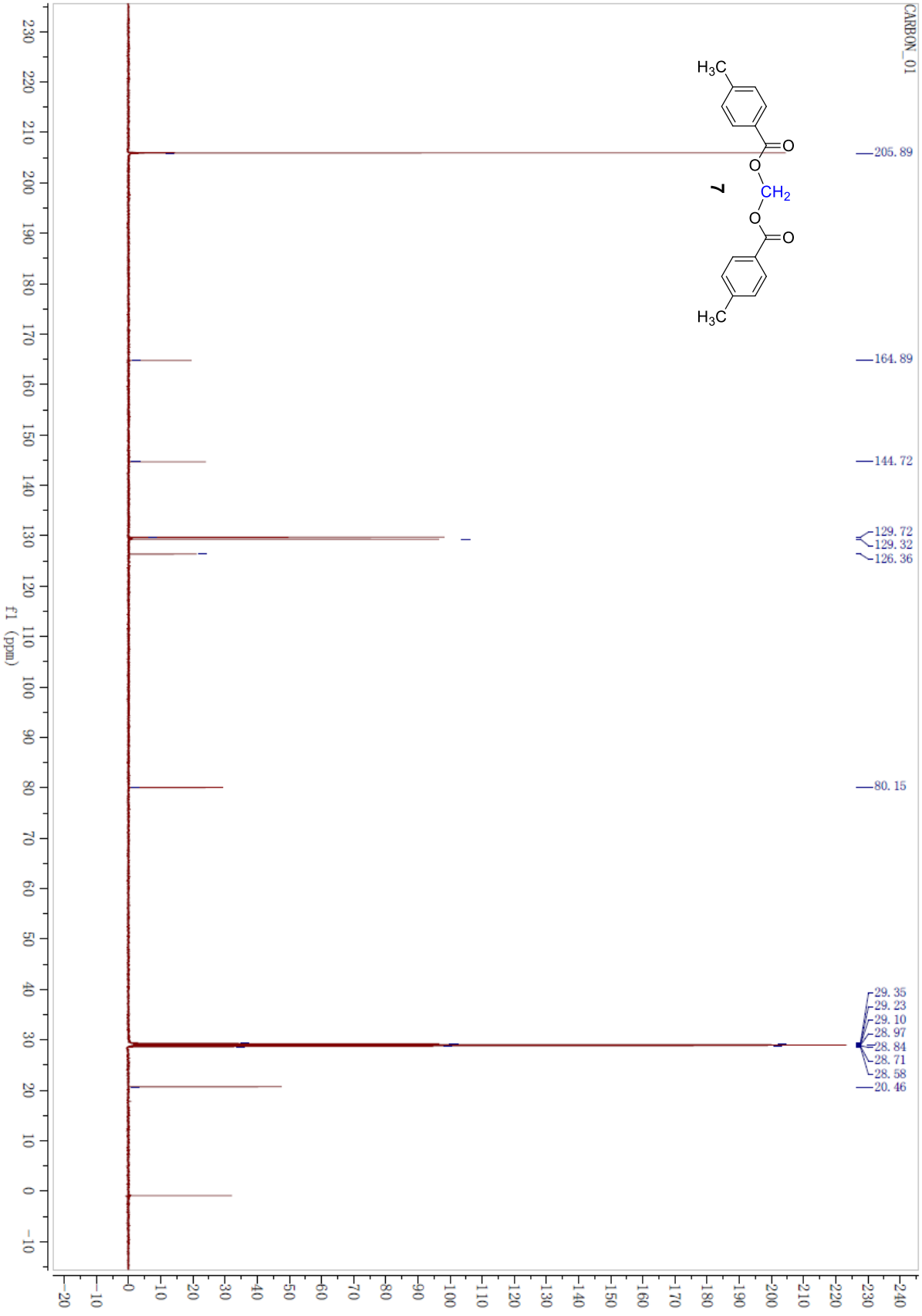

PROTON_01
f1 (ppm)
5
CH2
2.21
3.13
1.93
0.40
2.83
4.00
1.66
1.78
2.03
8.13
8.03
8.01
8.00
7.99
7.76
7.74
7.60
7.59
7.58
7.49
7.48
7.45
7.44
7.43
4.54
1.79
1.77
1.76
1.75
1.73
1.42
1.41
1.39
1.38
1.37
1.36
0.94
0.93
0.92

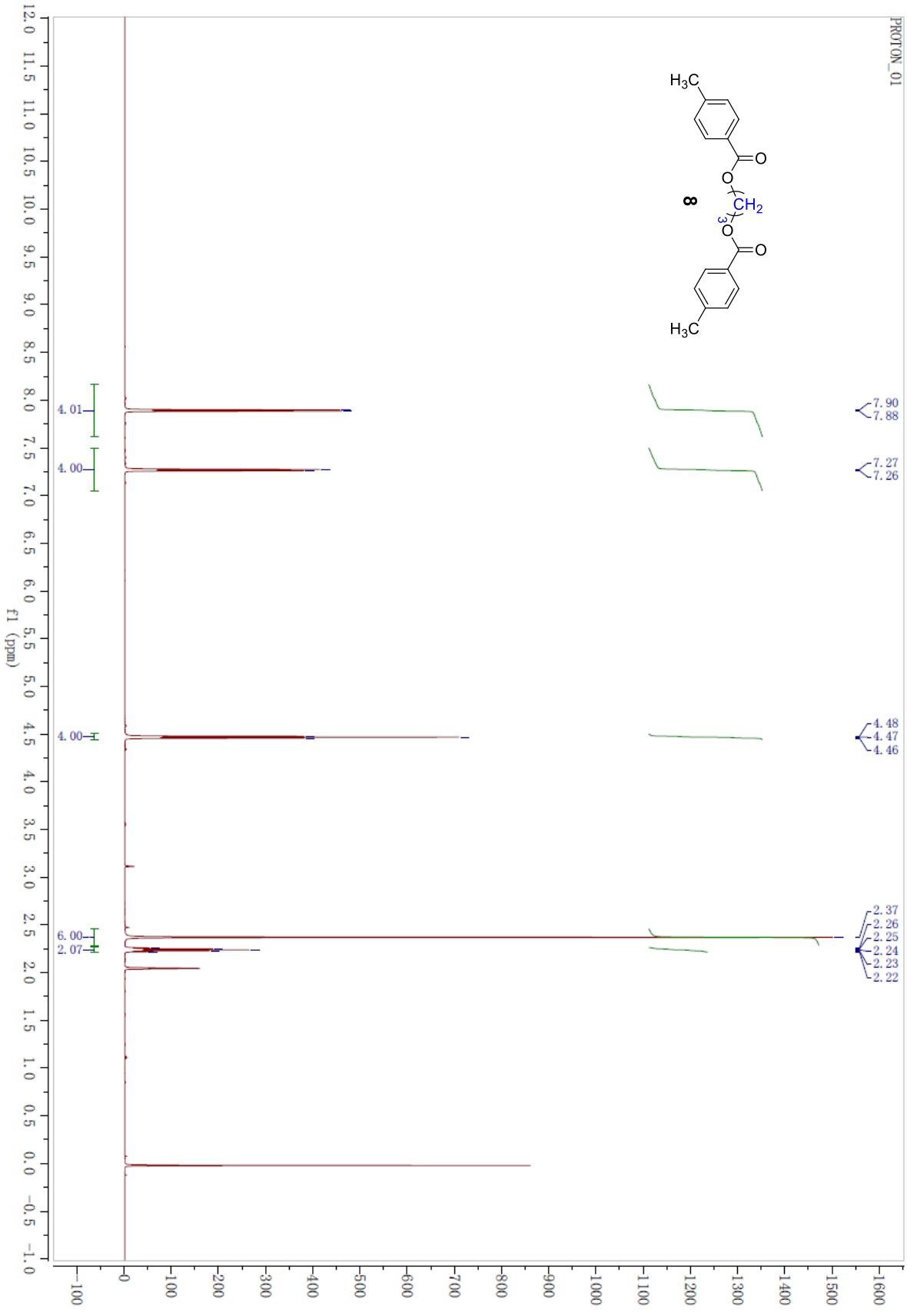

CARBON_01
205.90
166.39
132.79
130.73
129.47
129.20
128.49
64.25
25.36
f1 (ppm)
5

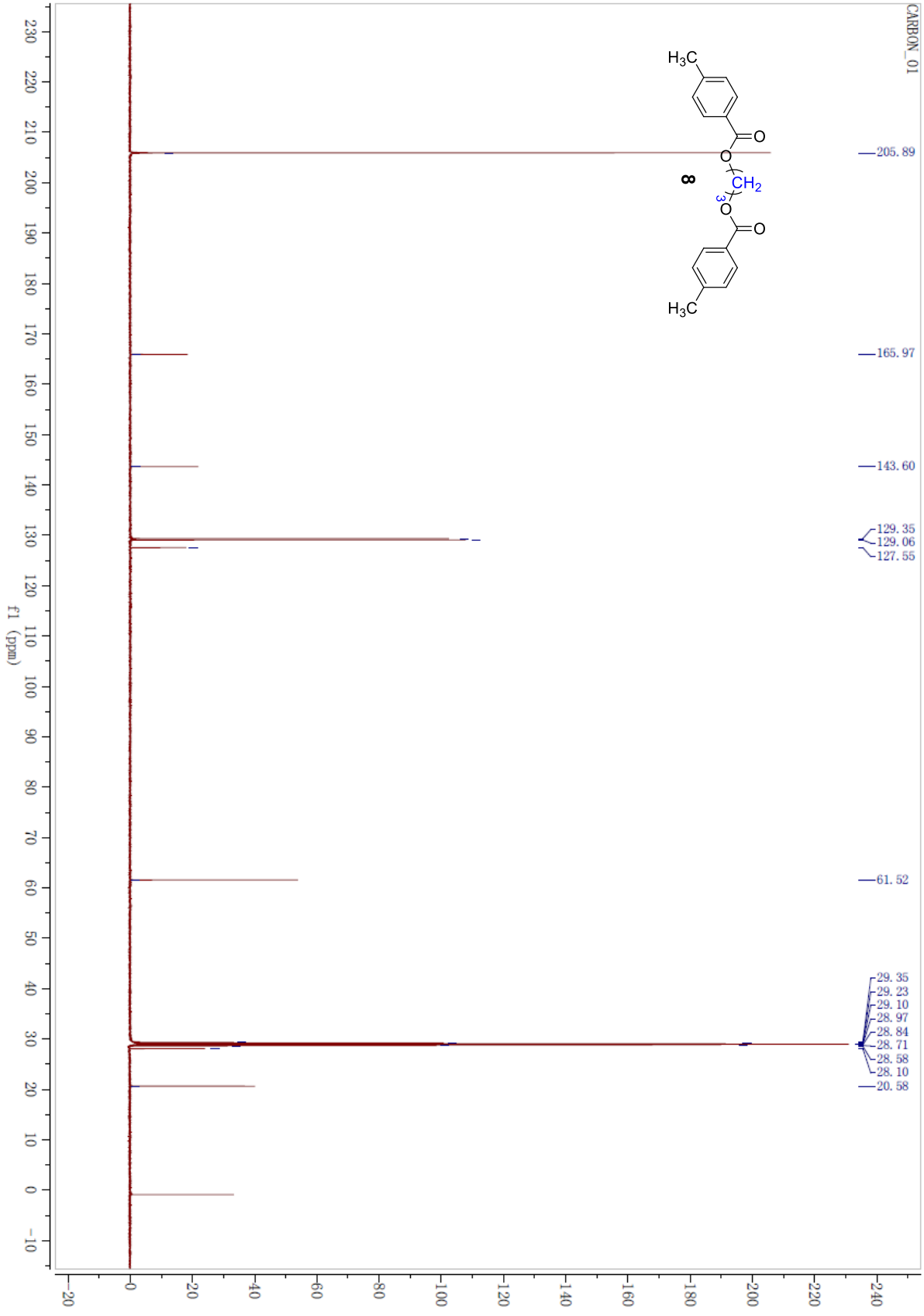

PROTON_01
f1 (ppm)
6
3.74
1.93
3.62
4.00
4.10
4.06
8.01
8.00
7.62
7.61
7.60
7.50
7.49
7.47
4.33
4.32
4.30
1.81
1.80
1.79
1.56
1.55
1.54

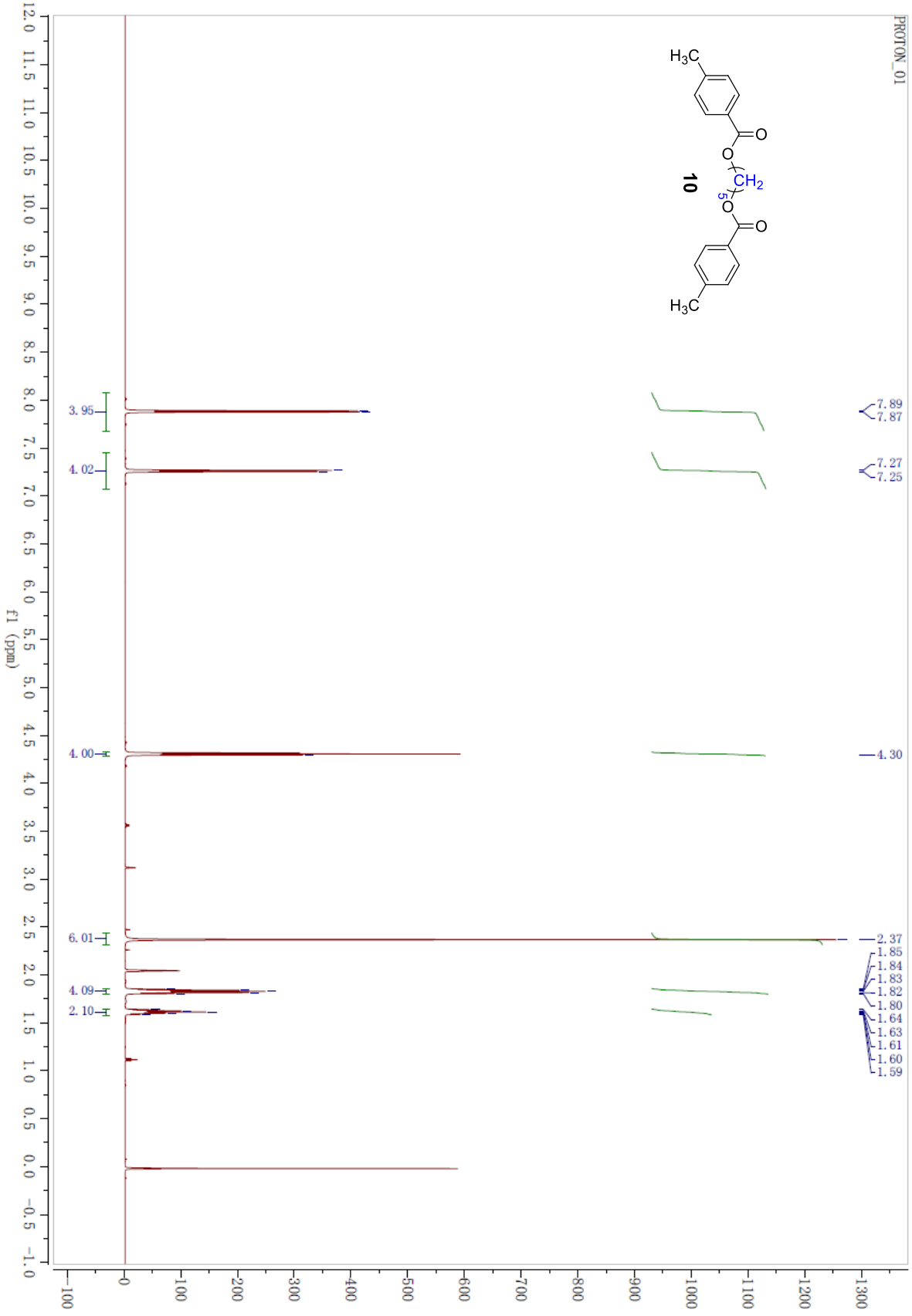

CARBON_01
205.90
166.00
132.91
130.50
129.21
128.48
64.53
25.54
f1 (ppm)
6
CH2

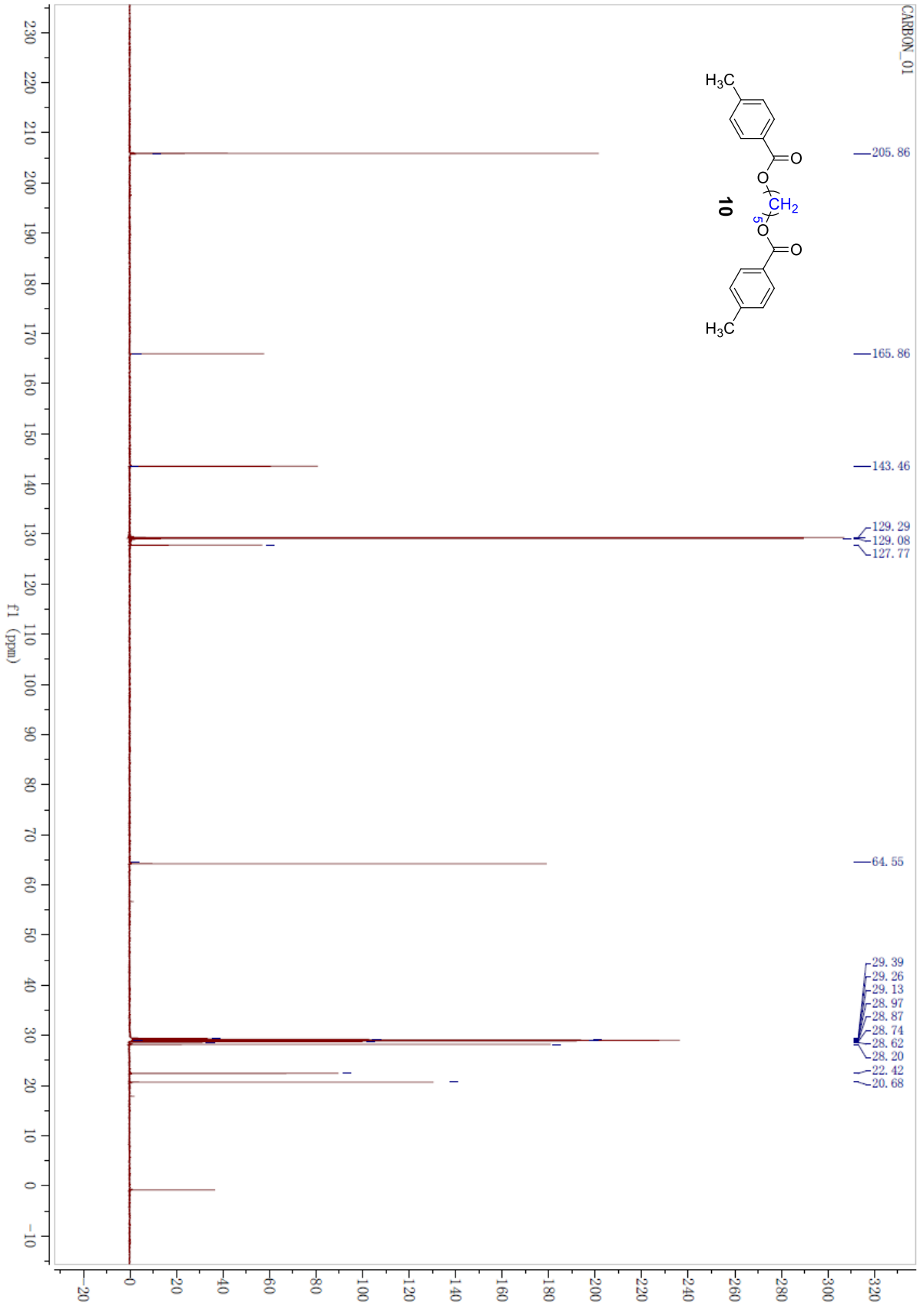

PROTON_01
f1 (ppm)
3.72
3.83
2.00
6.01
7.96
7.94
7.35
7.34
6.24
2.40
H3C
O
O
CH2
O
O
H3C
7

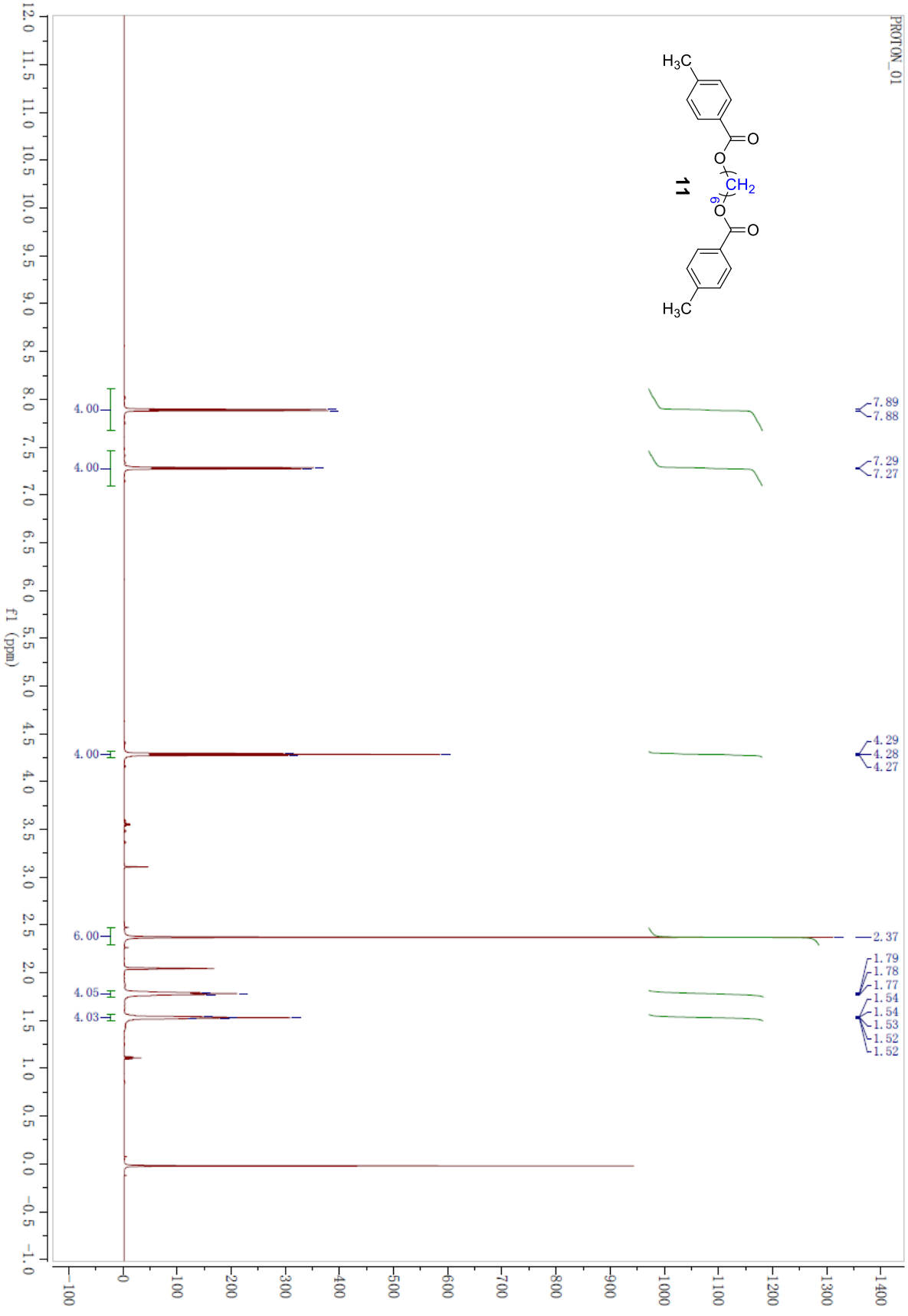

CARBON_01
205.89
164.89
144.72
129.72
129.32
126.36
80.15
29.35
29.23
29.10
28.97
28.84
28.71
28.58
20.46
f1 (ppm)
7

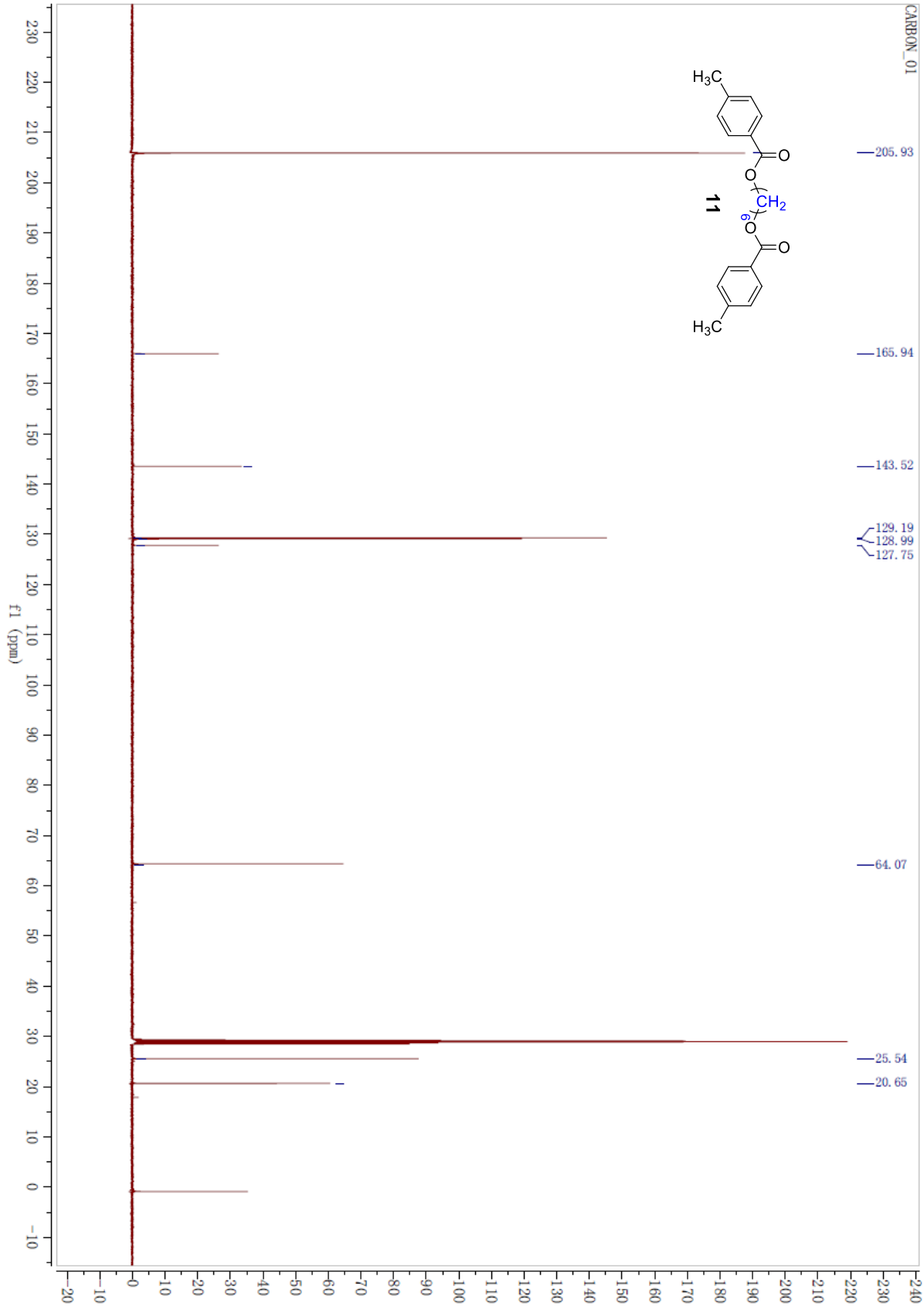

PROTON_01
f1 (ppm)
8
4.01
4.00
4.00
6.00
2.07
7.90
7.88
7.27
7.26
4.48
4.47
4.46
2.37
2.26
2.25
2.24
2.23
2.22

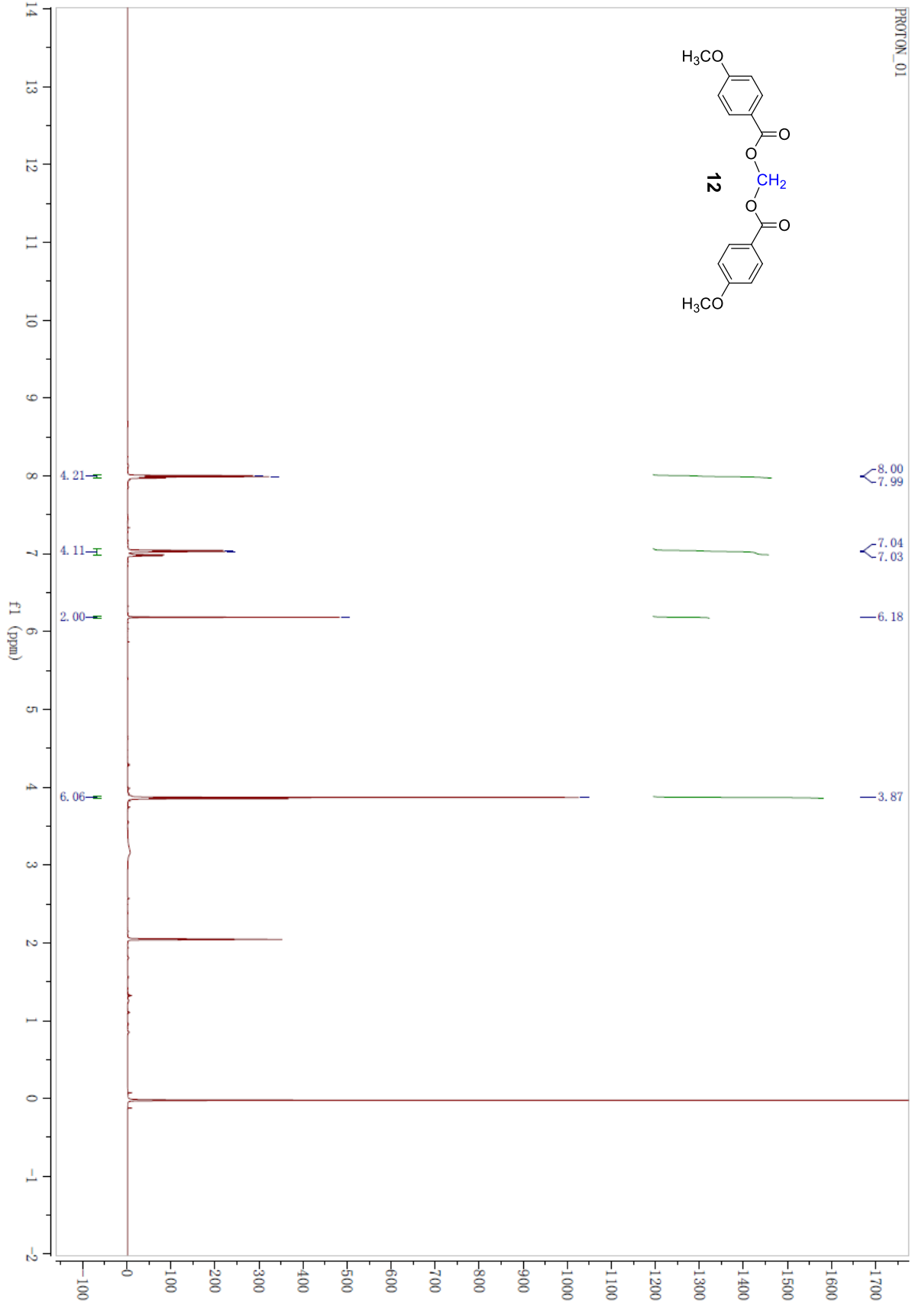

CARBON_01
205.89
165.97
143.60
129.35
129.06
127.55
61.52
29.35
29.23
29.10
28.97
28.84
28.71
28.58
28.10
20.58
f1 (ppm)
8
3

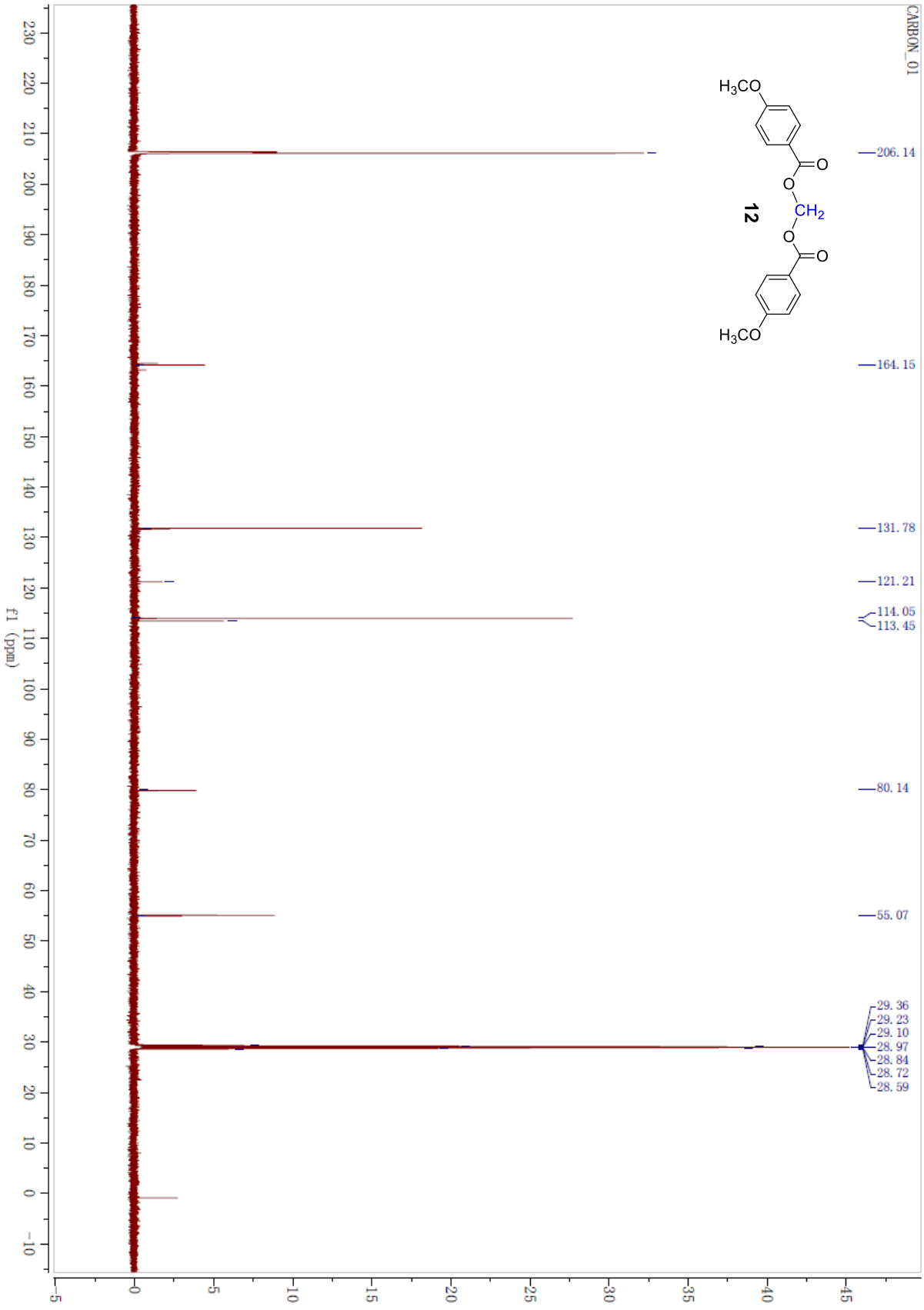

PROTON_01
f1 (ppm)
10
3.95
4.02
4.00
6.01
4.09
2.10
7.89
7.87
7.27
7.25
4.30
2.37
1.85
1.84
1.83
1.82
1.80
1.64
1.63
1.61
1.60
1.59

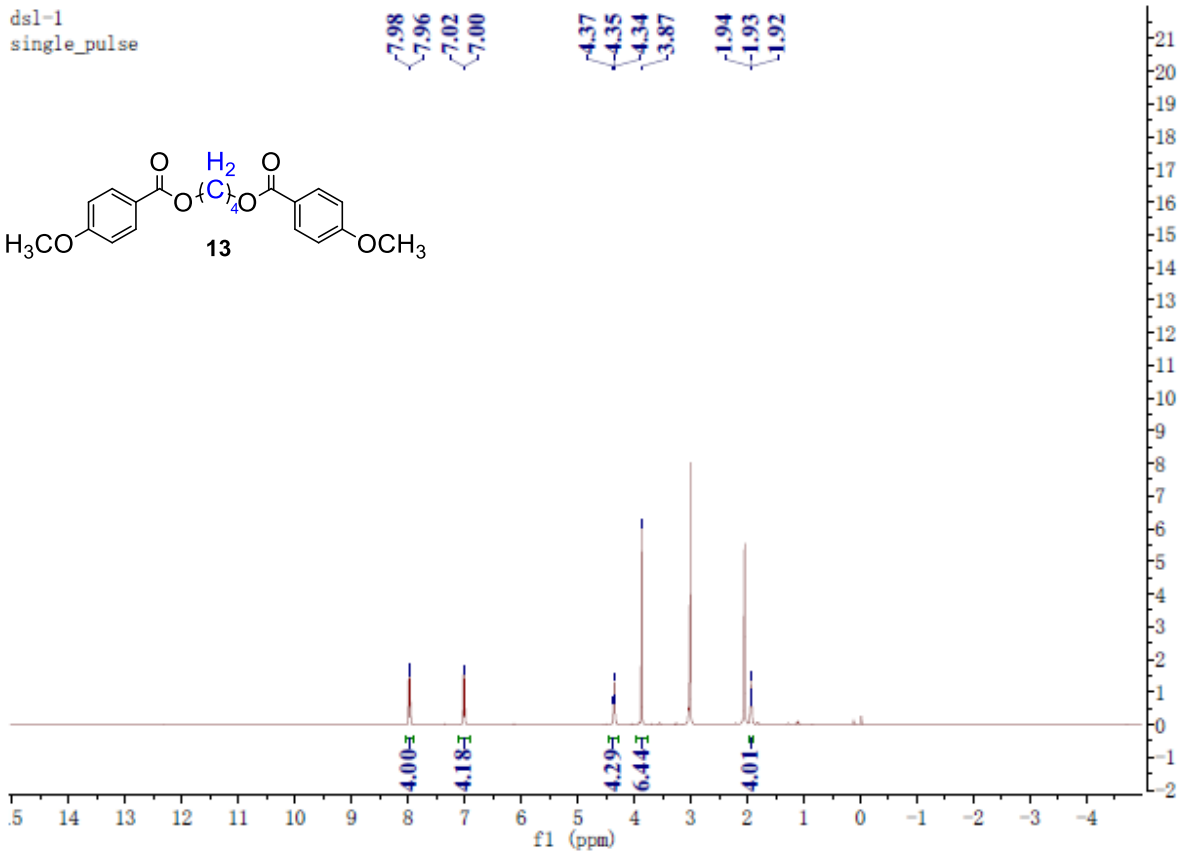

CARBON_01
205.86
165.86
143.46
129.29
129.08
127.77
64.55
29.39
29.26
29.13
28.97
28.87
28.74
28.62
28.20
22.42
20.68
10
f1 (ppm)

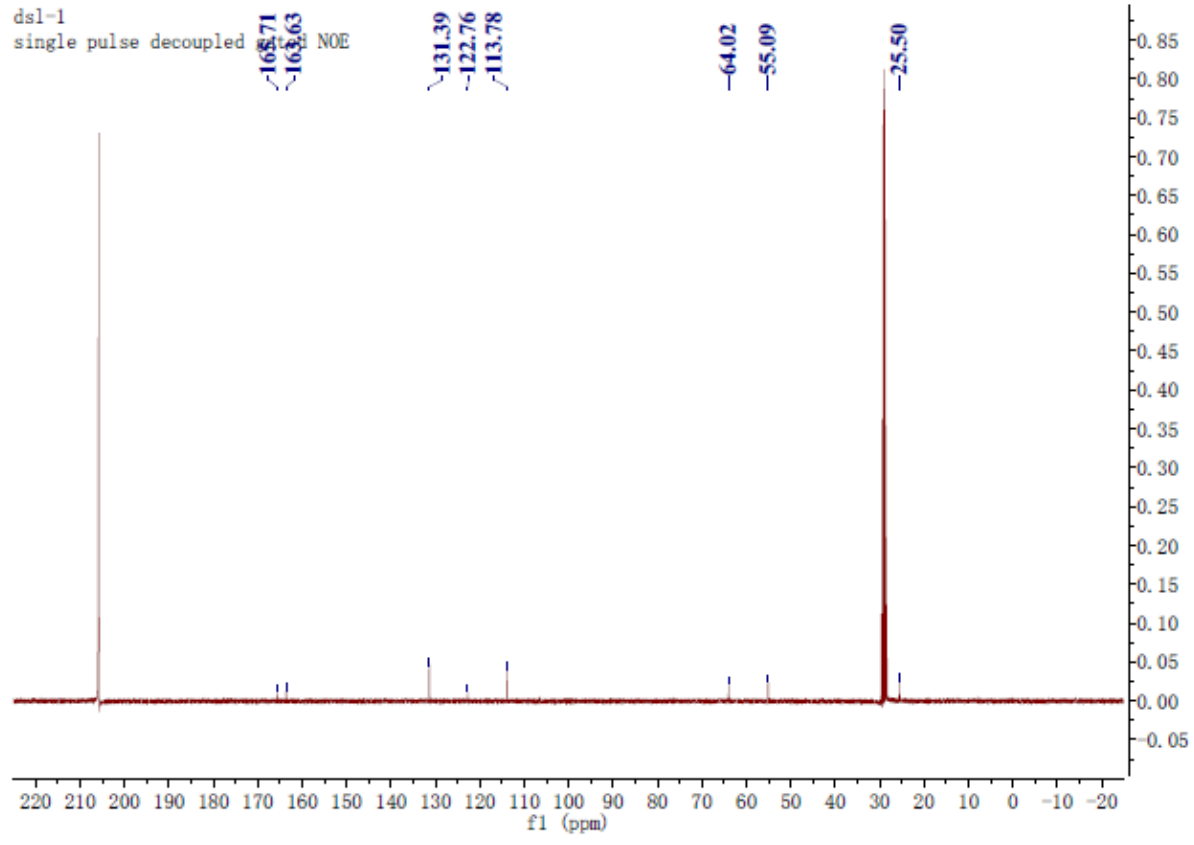

PROTON_01
f1 (ppm)
11
4.00
4.00
4.00
6.00
4.05
4.03
7.89
7.88
7.29
7.27
4.29
4.28
4.27
2.37
1.79
1.78
1.77
1.54
1.54
1.53
1.52
1.52


CARBON_01
205.93
165.94
143.52
129.19
128.99
127.75
64.07
25.54
20.65
f1 (ppm)
11

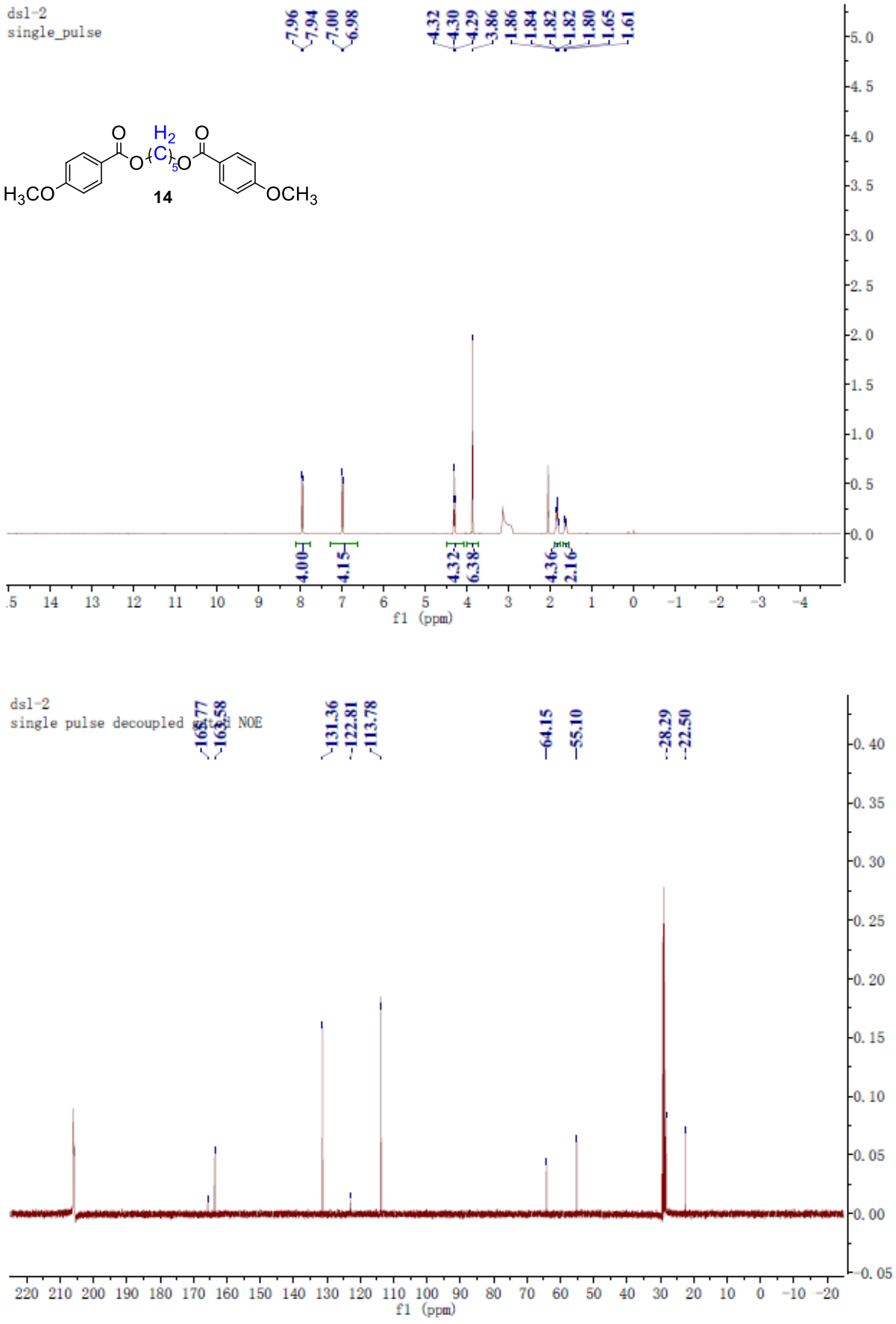

PROTON_01
12
H_3CO
O
O
CH_2
O
O
H_3CO
4.21
4.11
2.00
6.06
8.00
7.99
7.04
7.03
6.18
3.87
f1 (ppm)


CARBON_01
206.14
164.15
131.78
121.21
114.05
113.45
80.14
55.07
29.36
29.23
29.10
28.97
28.84
28.72
28.59
f1 (ppm)
12
H3CO
CH2
O
H3CO

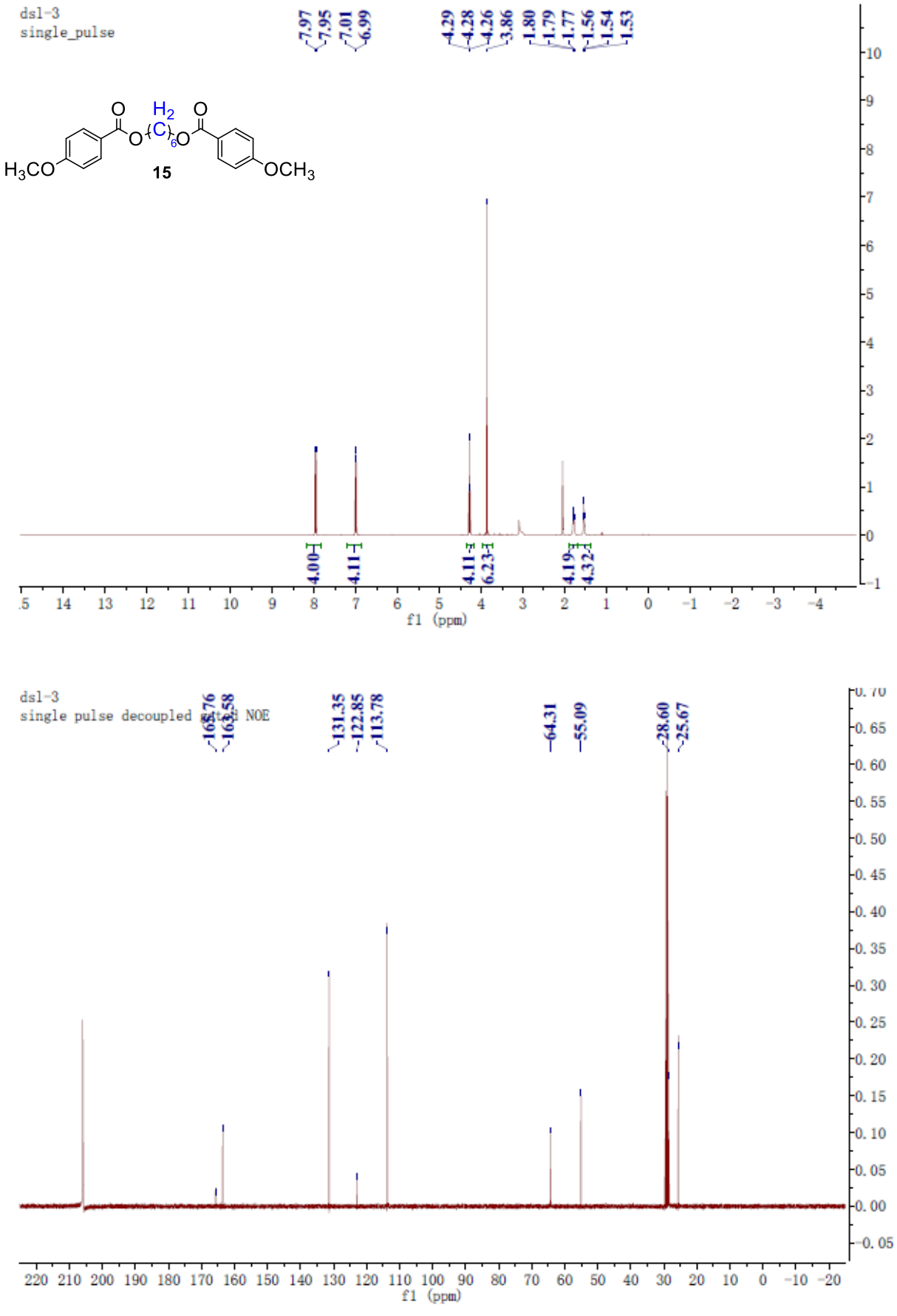




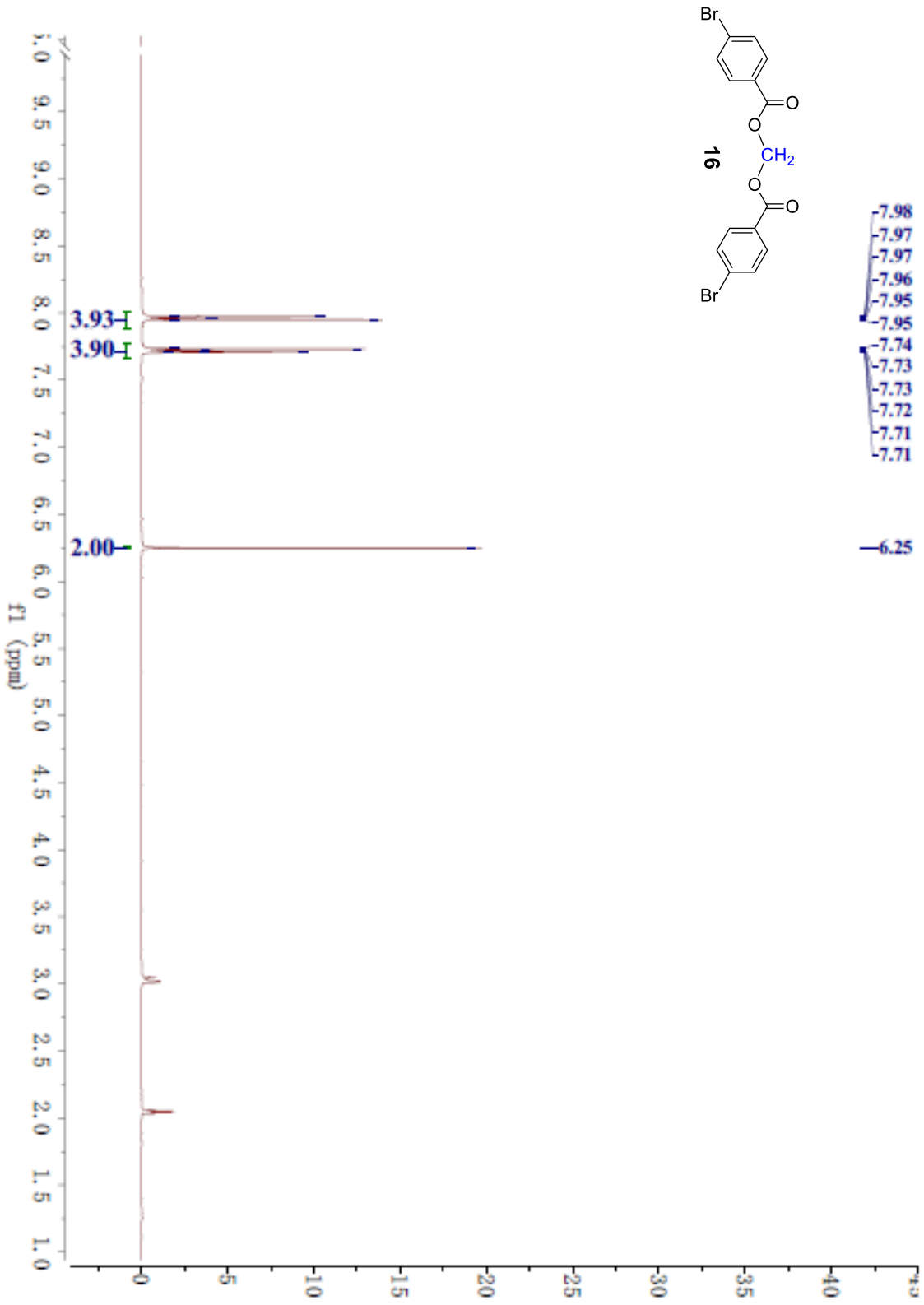
^{1}H and ^{13}C NMR Spectra of compound **13**

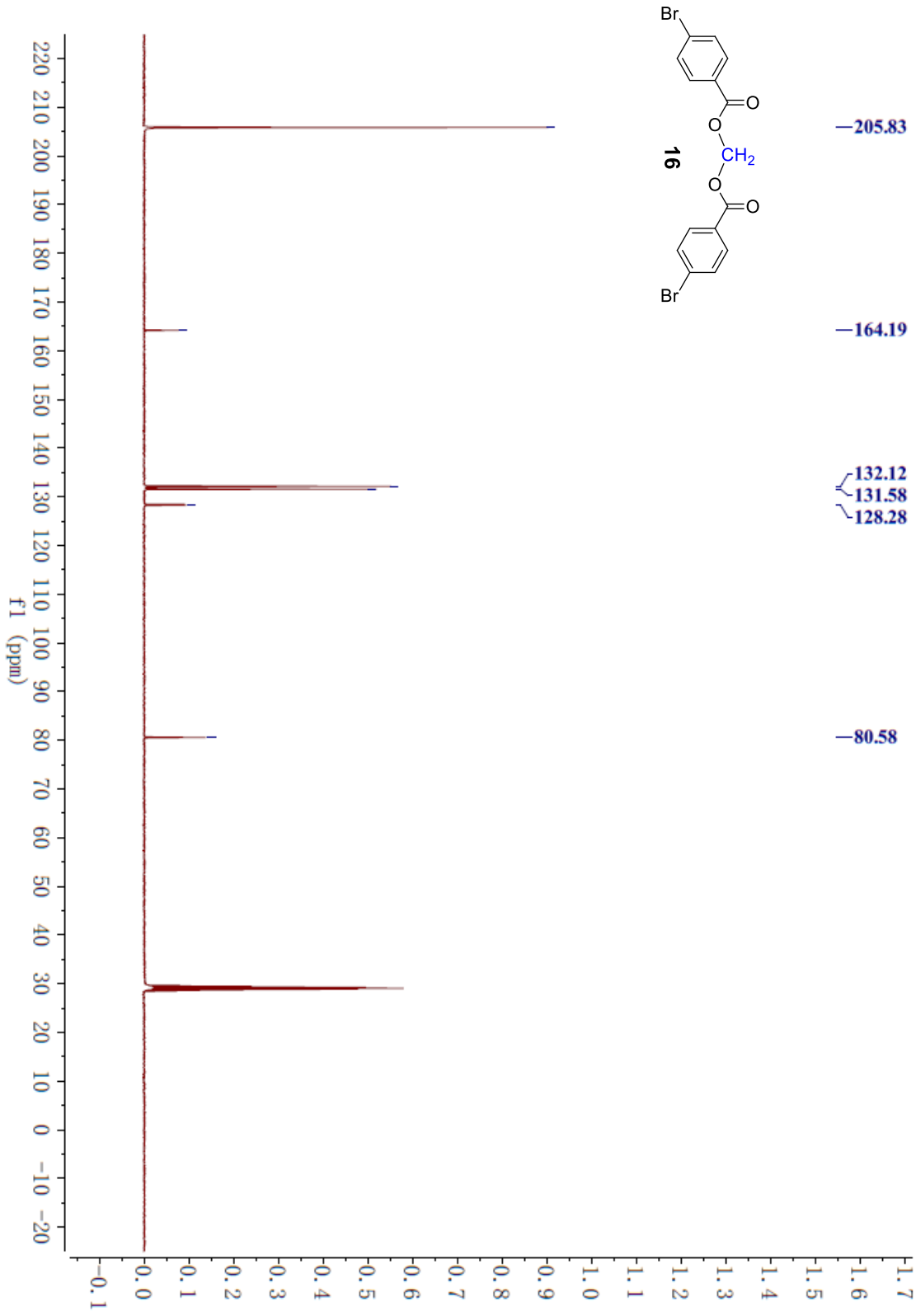


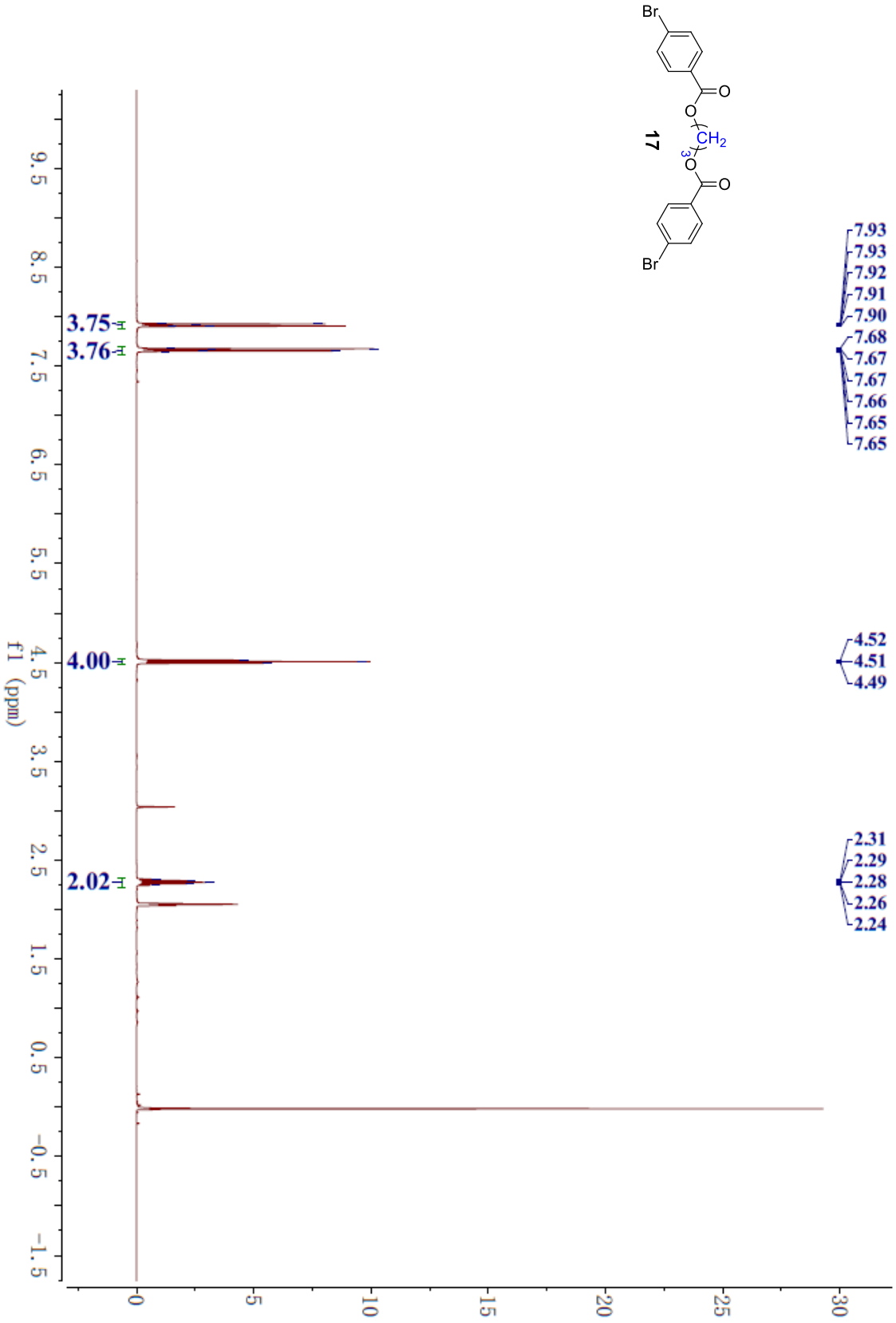
^{1}H and ^{13}C NMR Spectra of compound **14**

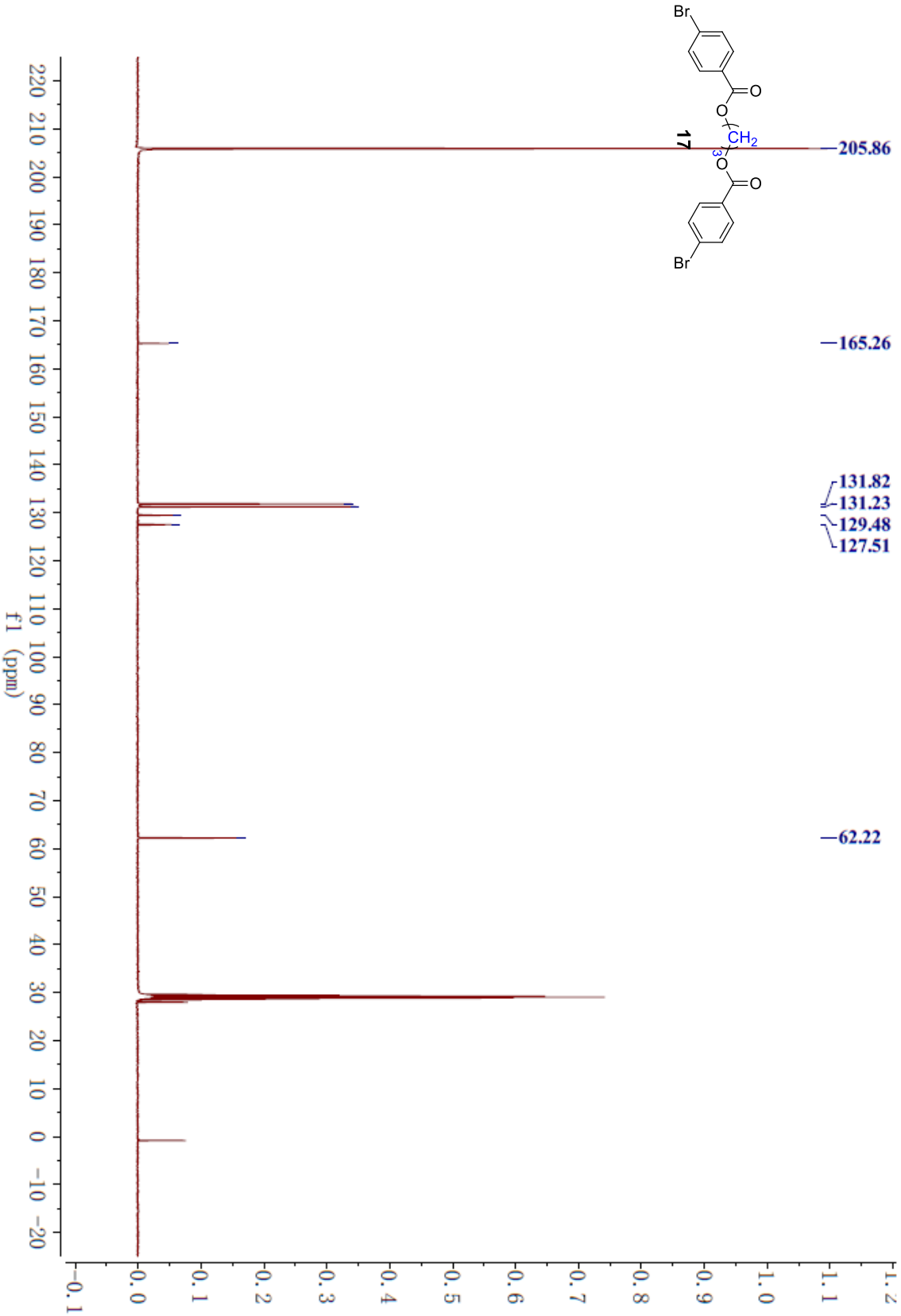


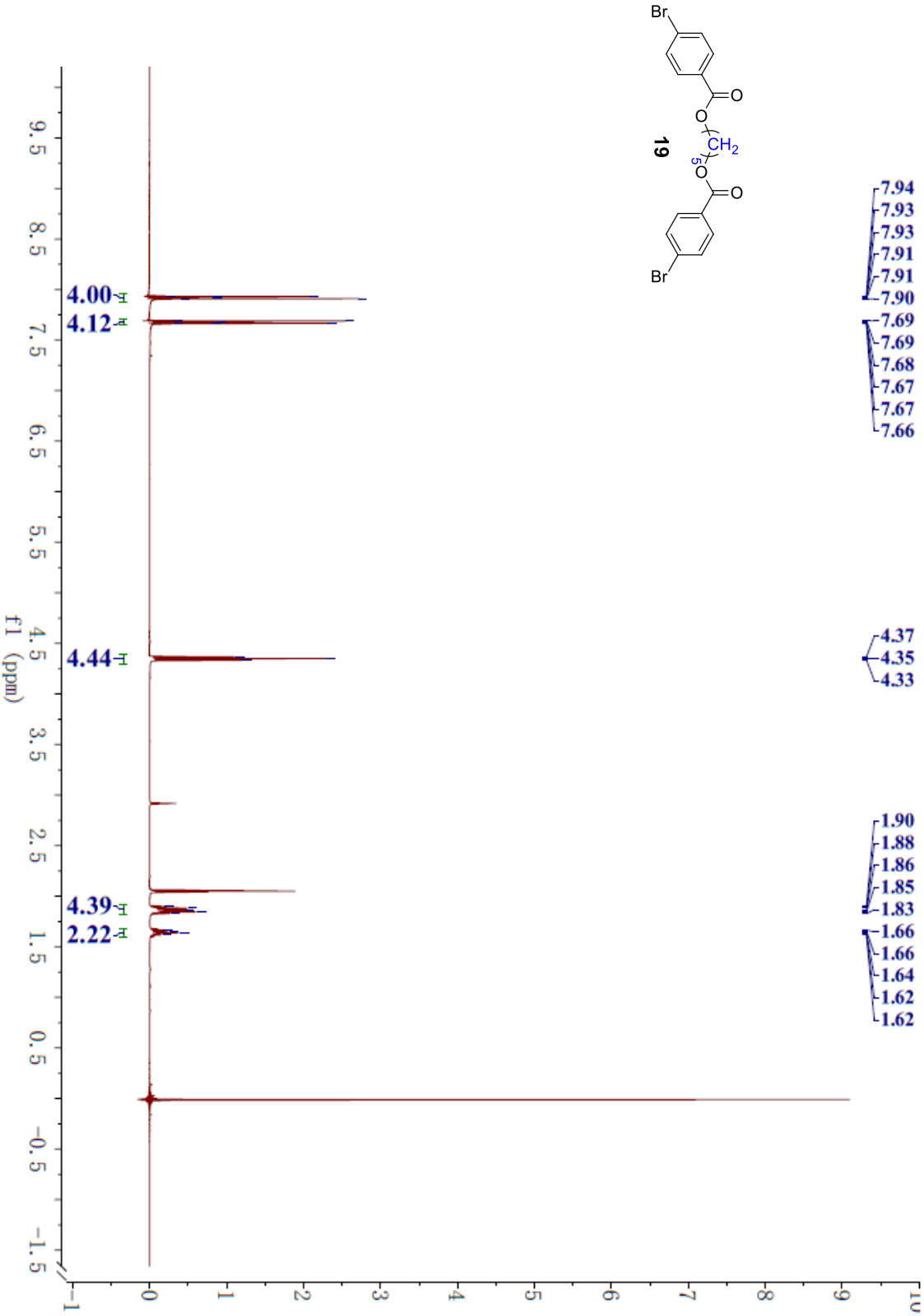
^{1}H and ^{13}C NMR Spectra of compound **15**

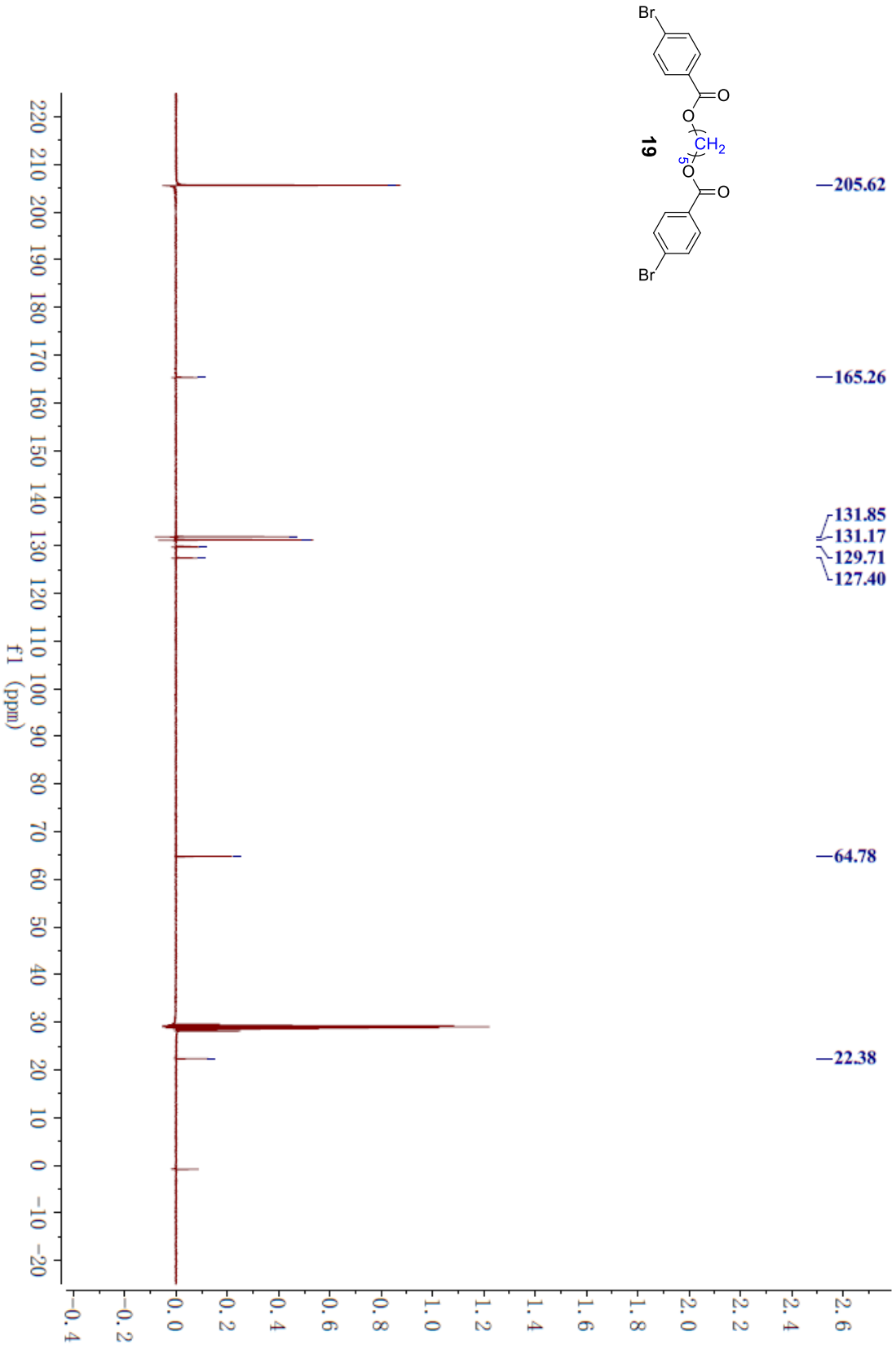

Br
O
O
CH2
O
O
Br
16
7.98
7.97
7.97
7.96
7.95
7.95
7.74
7.73
7.73
7.72
7.71
7.71
6.25
3.93
3.90
2.00
f1 (ppm)

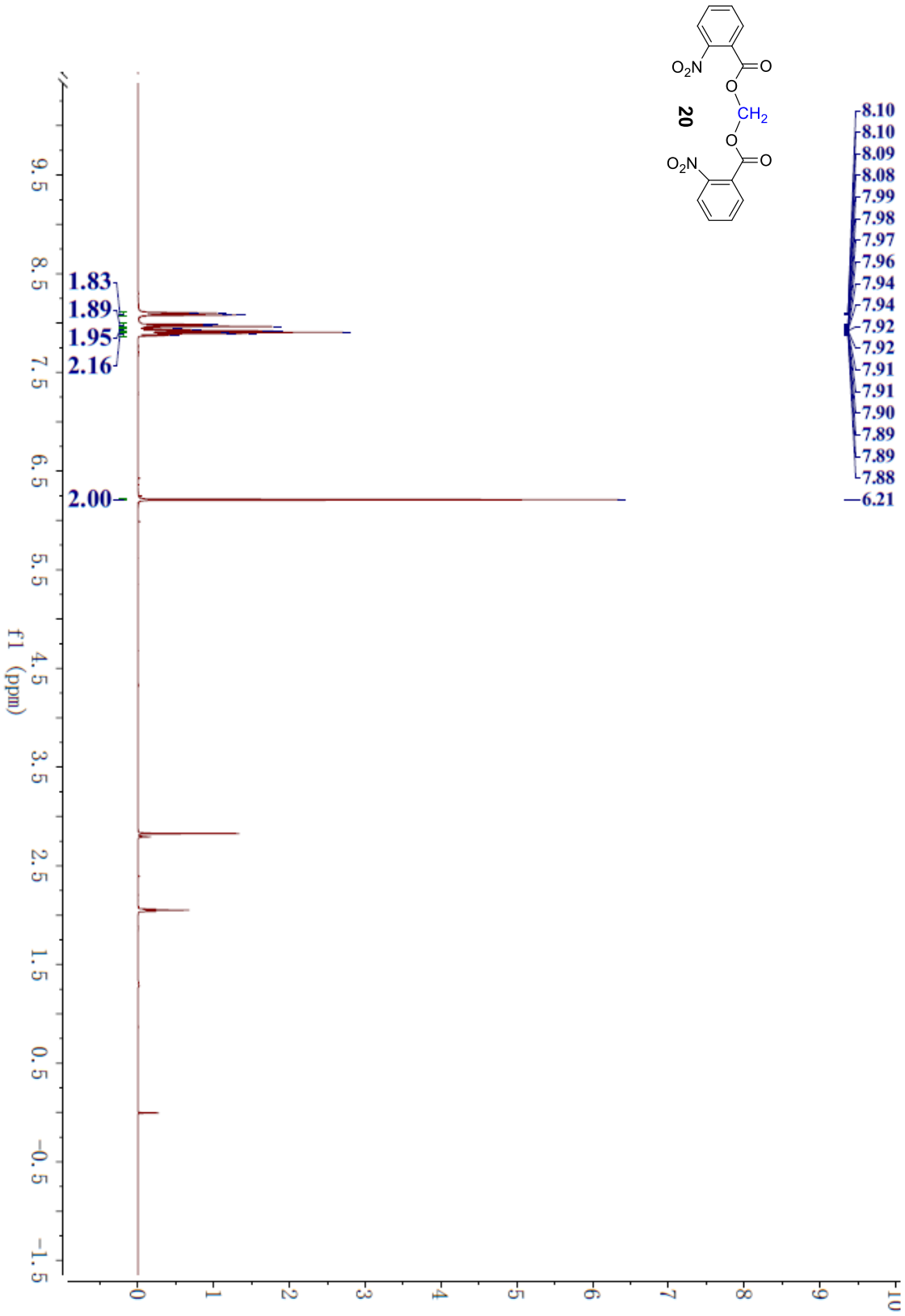

Br
O
O
CH2
O
O
Br
16
205.83
164.19
132.12
131.58
128.28
80.58
f1 (ppm)

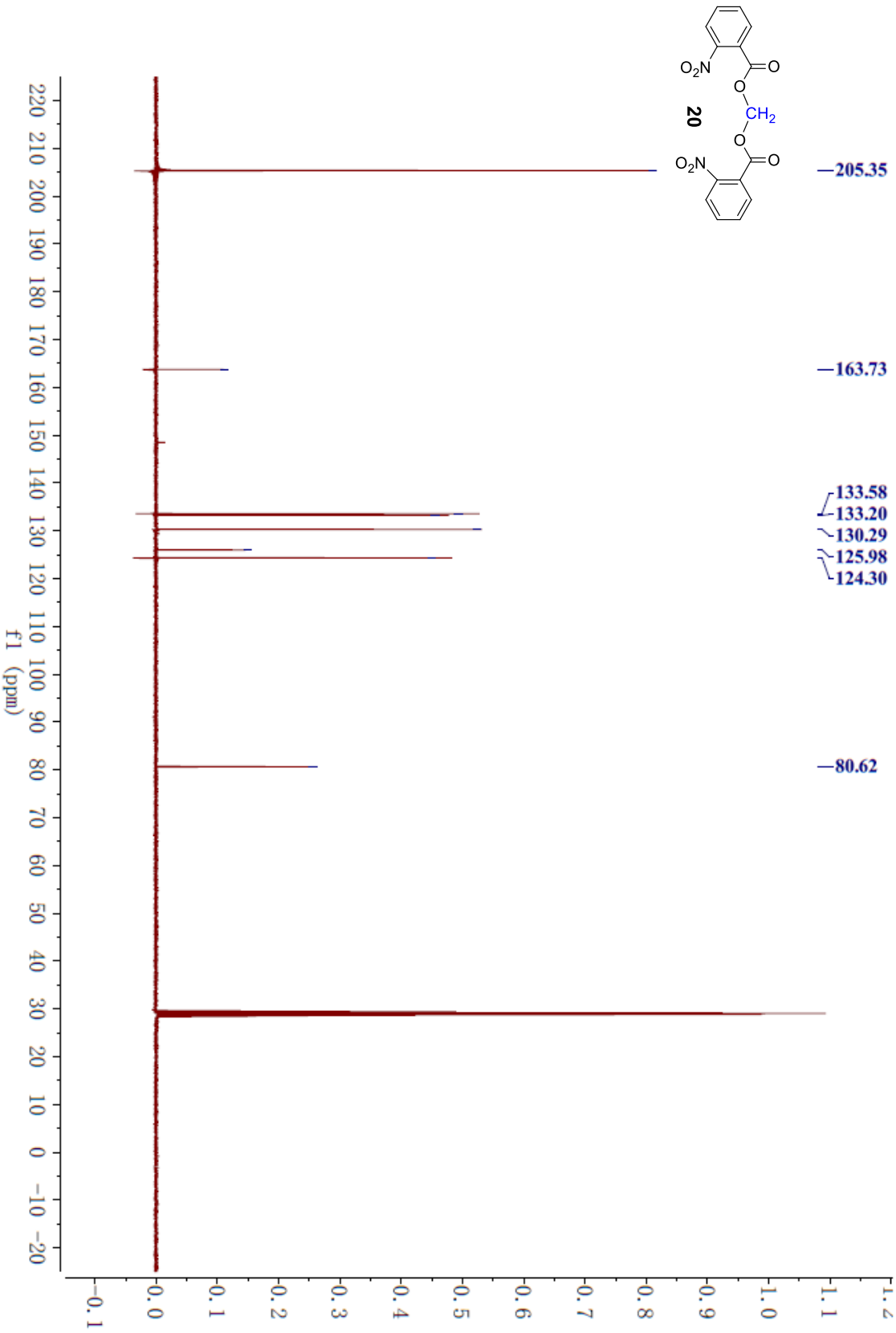

Br
O
O
CH2
3
O
O
Br
17
7.93
7.93
7.92
7.91
7.90
7.68
7.67
7.67
7.66
7.65
7.65
4.52
4.51
4.49
2.31
2.29
2.28
2.26
2.24
3.75
3.76
4.00
2.02
f1 (ppm)
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
−0.5
−1.5
0
5
10
15
20
25
30

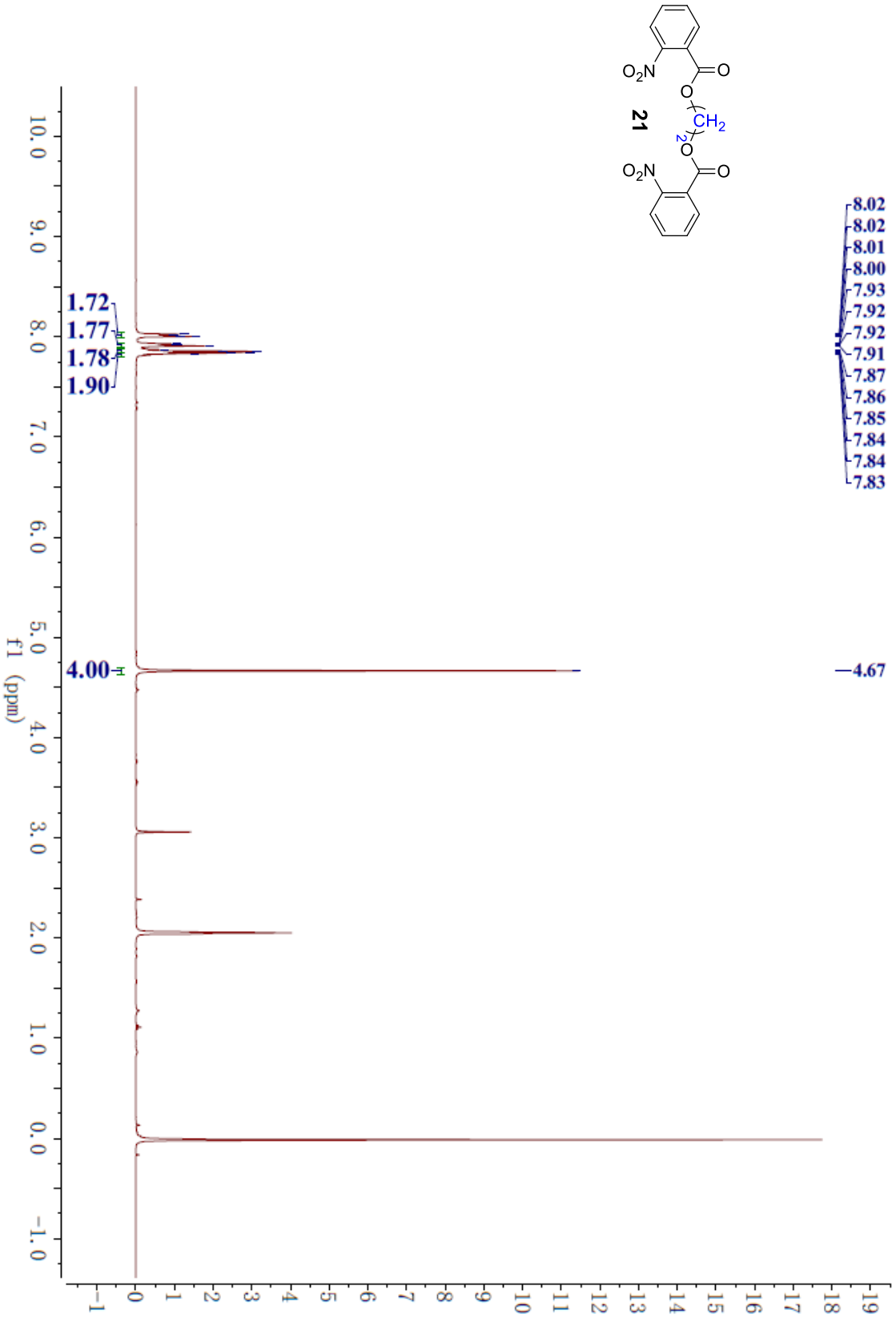

Br
O
O
CH2
3
O
O
Br
17
205.86
165.26
131.82
131.23
129.48
127.51
62.22
f1 (ppm)

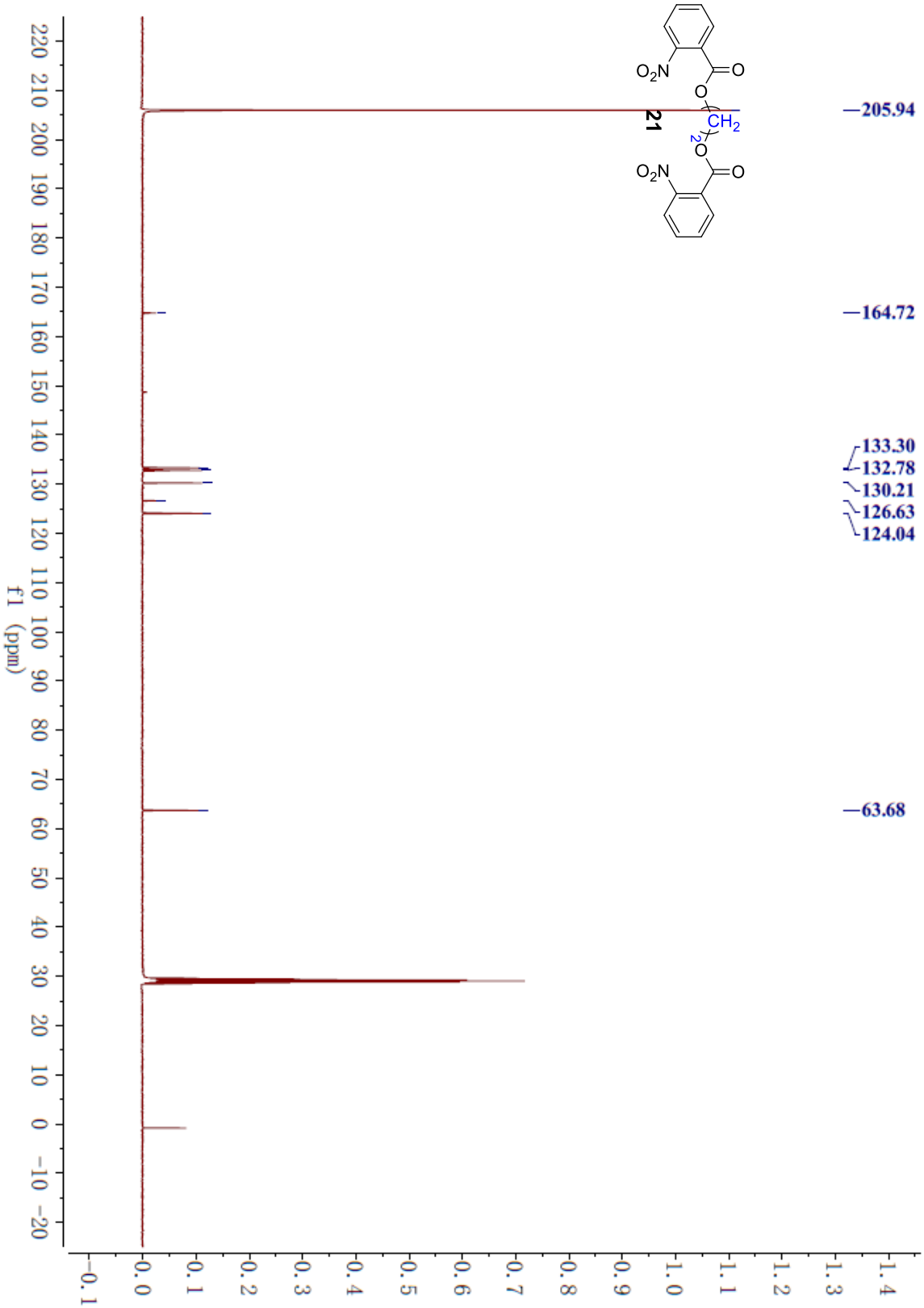

Br
O
O
CH2
5
O
O
Br
19
7.94
7.93
7.93
7.91
7.91
7.90
7.69
7.69
7.68
7.67
7.67
7.66
4.37
4.35
4.33
1.90
1.88
1.86
1.85
1.83
1.66
1.66
1.64
1.62
1.62
4.00
4.12
4.44
4.39
2.22
f1 (ppm)

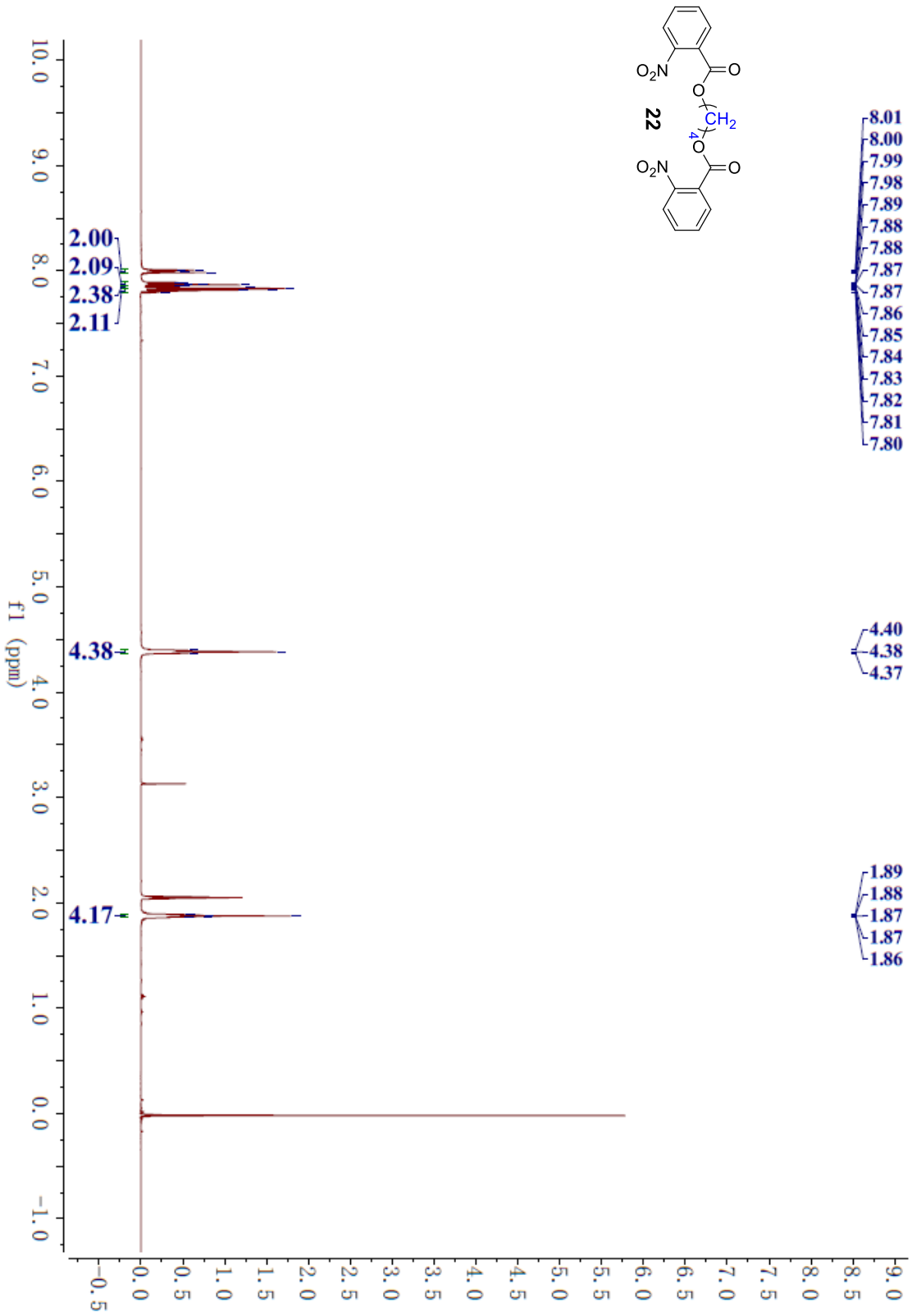

Br
O
O
CH2
O
O
Br
19
205.62
165.26
131.85
131.17
129.71
127.40
64.78
22.38
f1 (ppm)

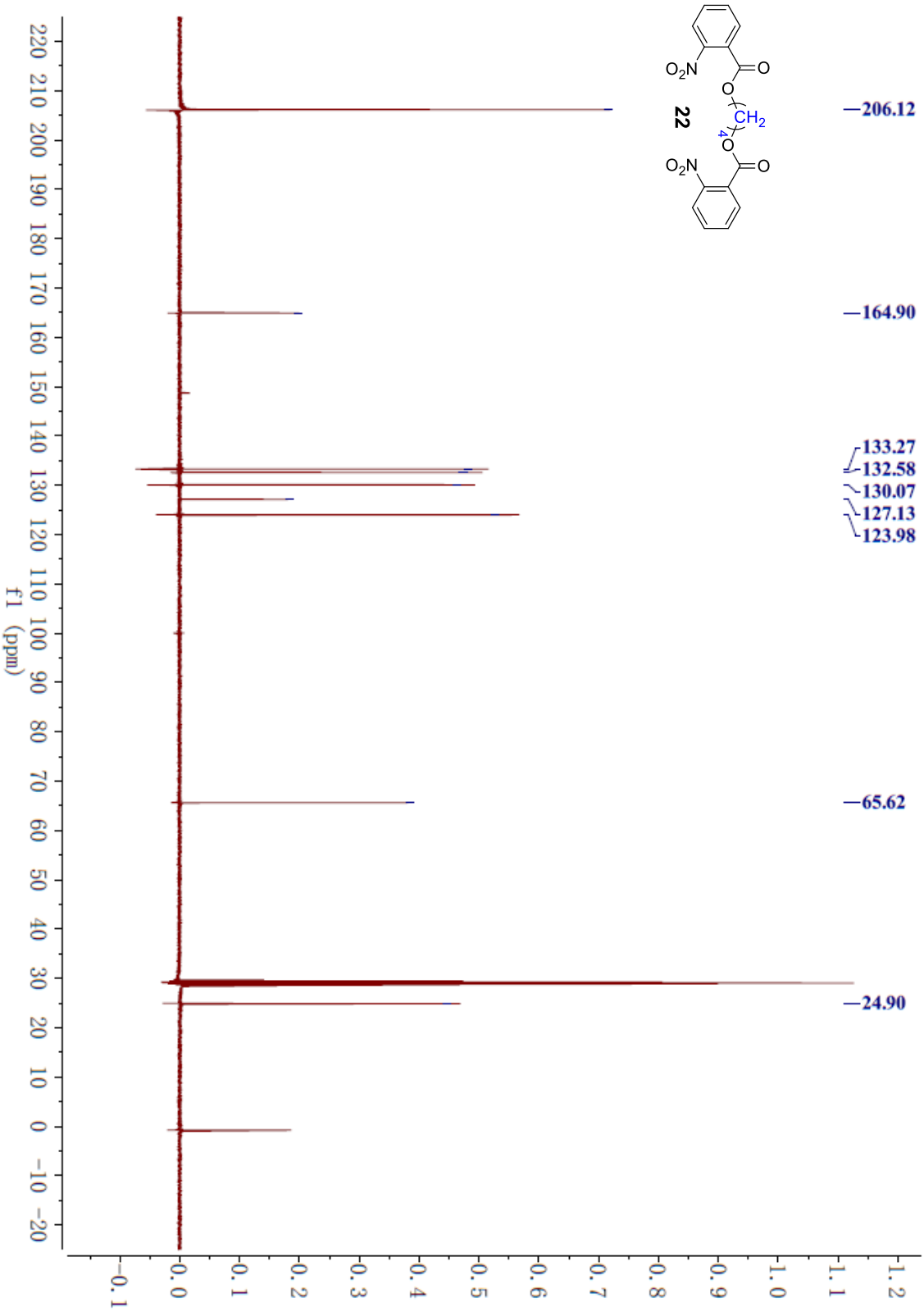

20
1.83
1.89
1.95
2.16
2.00
8.10
8.10
8.09
8.08
7.99
7.98
7.97
7.96
7.94
7.94
7.92
7.92
7.91
7.91
7.90
7.89
7.89
7.88
6.21
f1 (ppm)

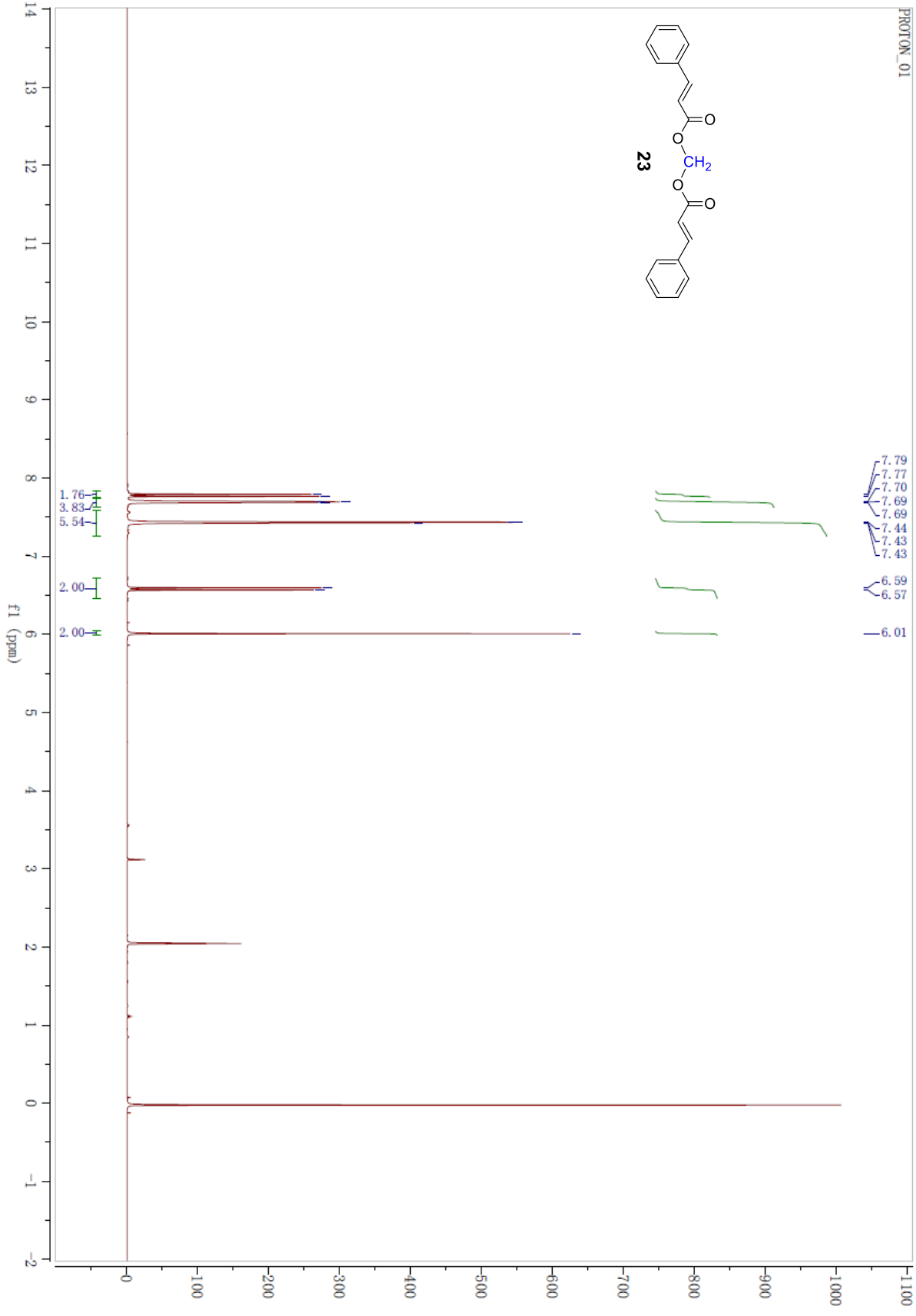

20
205.35
163.73
133.58
133.20
130.29
125.98
124.30
80.62
f1 (ppm)

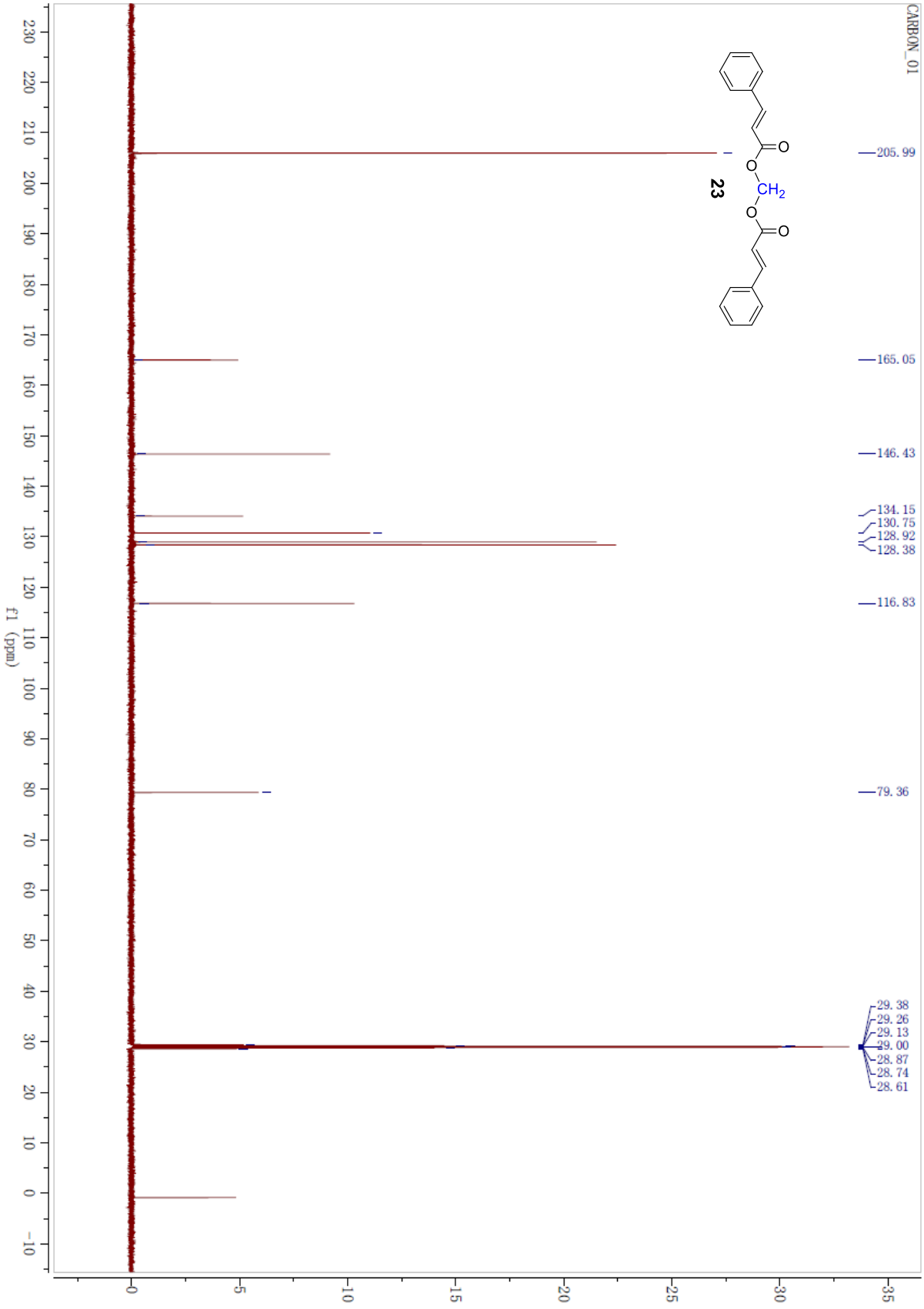

21
1.72
1.77
1.78
1.90
4.00
4.67
8.02
8.02
8.01
8.00
7.93
7.92
7.92
7.91
7.87
7.86
7.85
7.84
7.84
7.83
f1 (ppm)

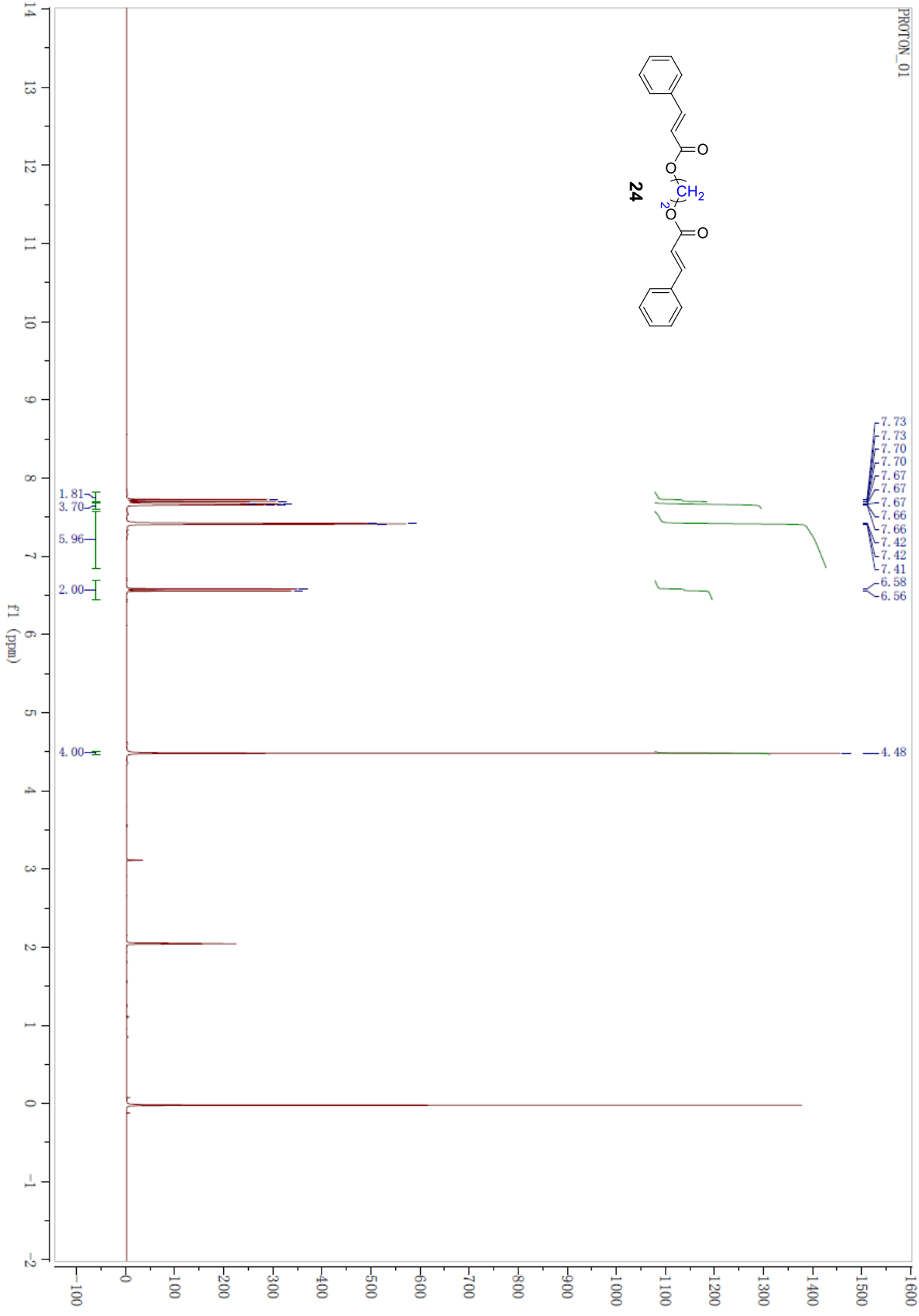

21
205.94
164.72
133.30
132.78
130.21
126.63
124.04
63.68
f1 (ppm)

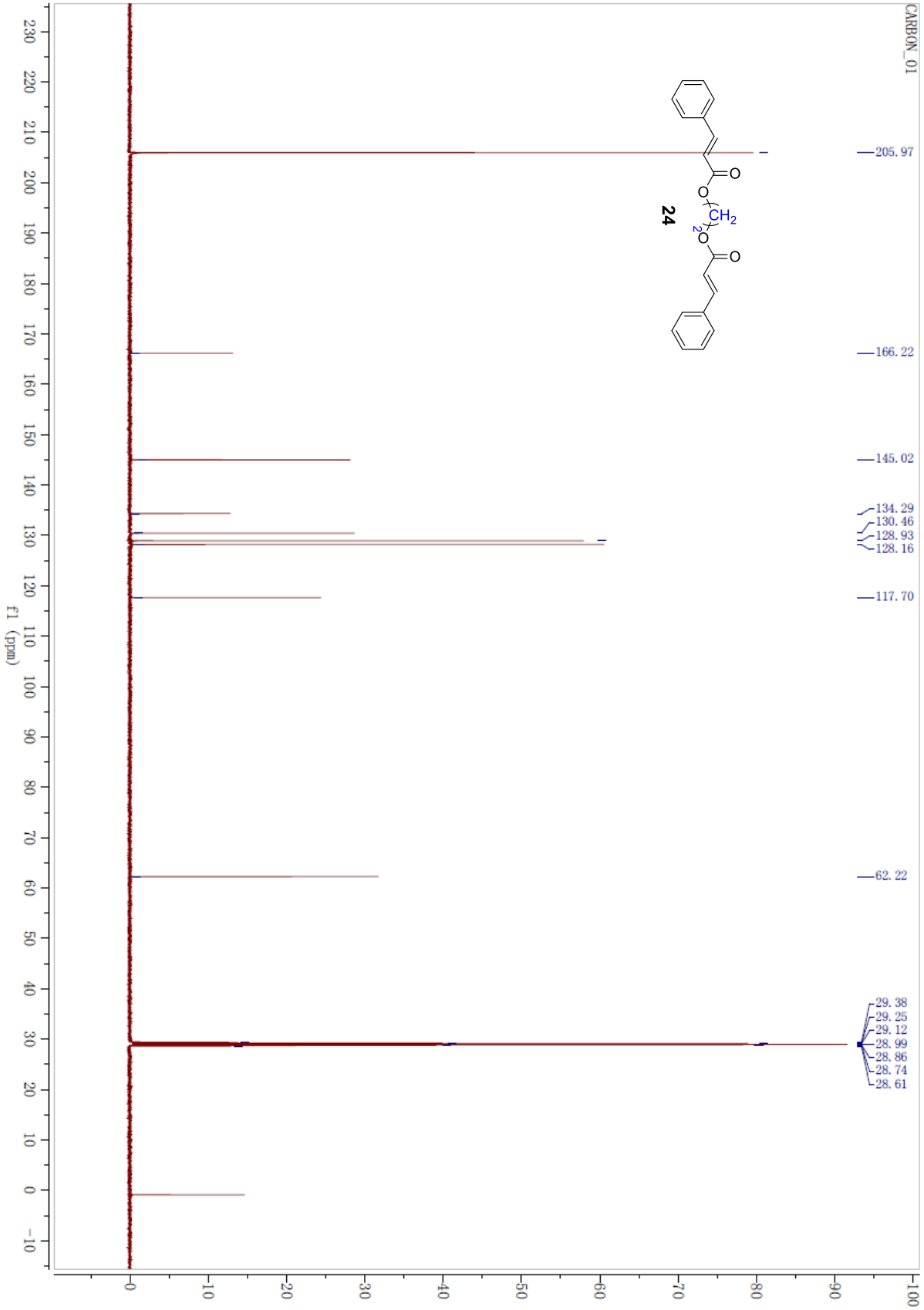

22
O2N
O
O
CH2
4
O
O2N
O
2.00
2.09
2.38
2.11
4.38
4.17
8.01
8.00
7.99
7.98
7.89
7.88
7.88
7.87
7.87
7.86
7.85
7.84
7.83
7.82
7.81
7.80
4.40
4.38
4.37
1.89
1.88
1.87
1.87
1.86
f1 (ppm)

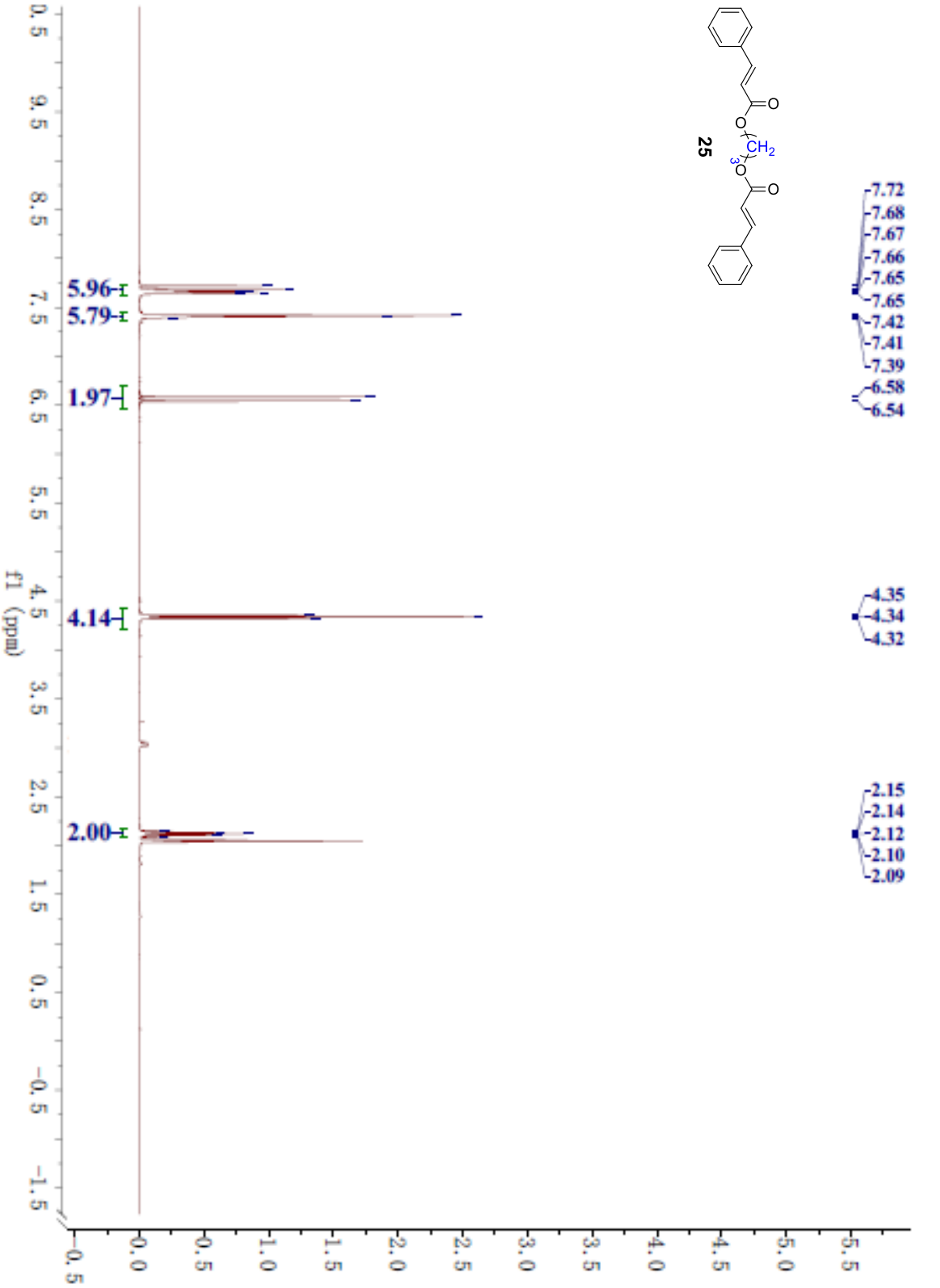

22
206.12
164.90
133.27
132.58
130.07
127.13
123.98
65.62
24.90
f1 (ppm)

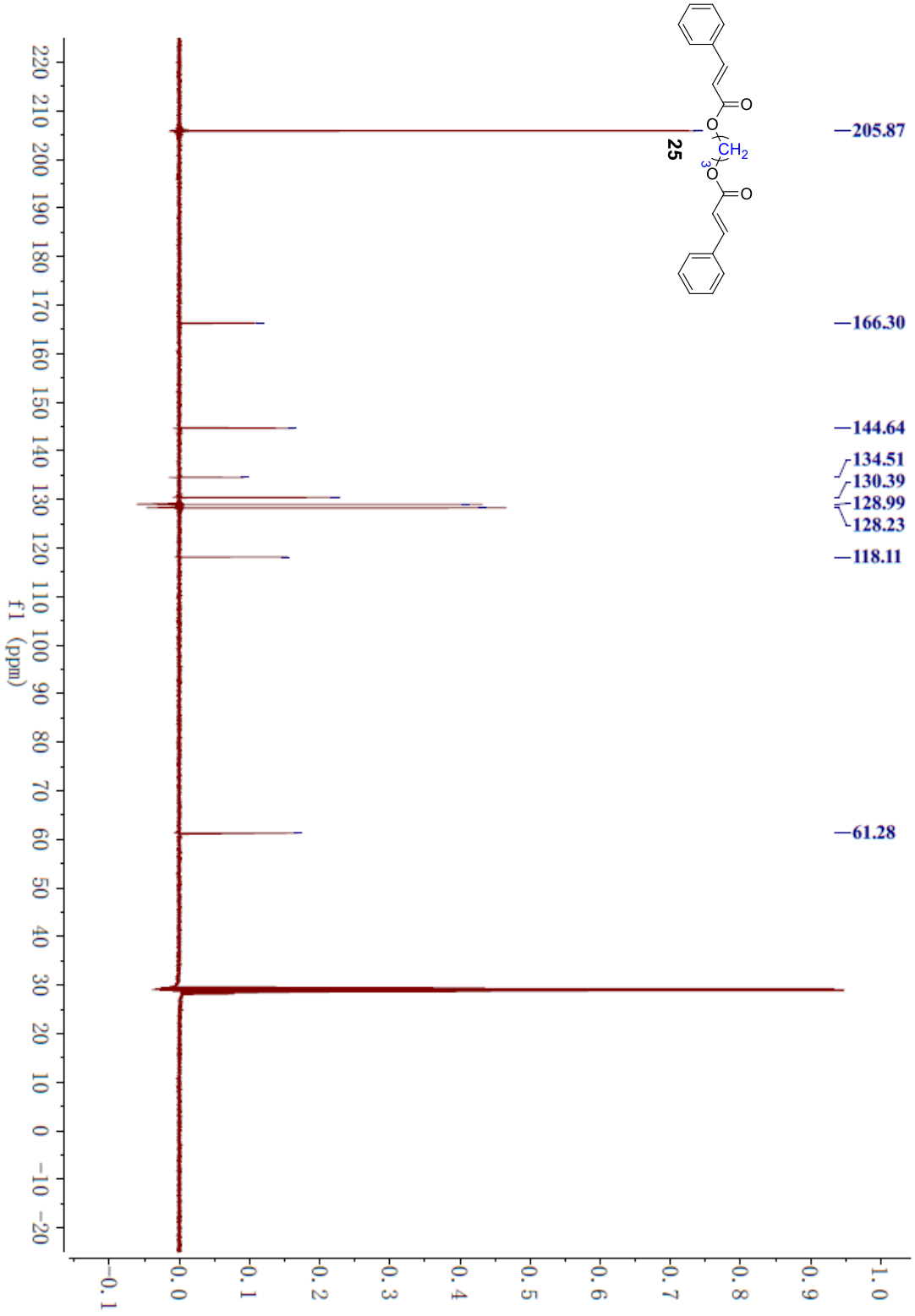

PROTON_01
23
O
O
CH2
O
O
1.76
3.83
5.54
2.00
2.00
7.79
7.77
7.70
7.69
7.69
7.44
7.43
7.43
6.59
6.57
6.01
f1 (ppm)

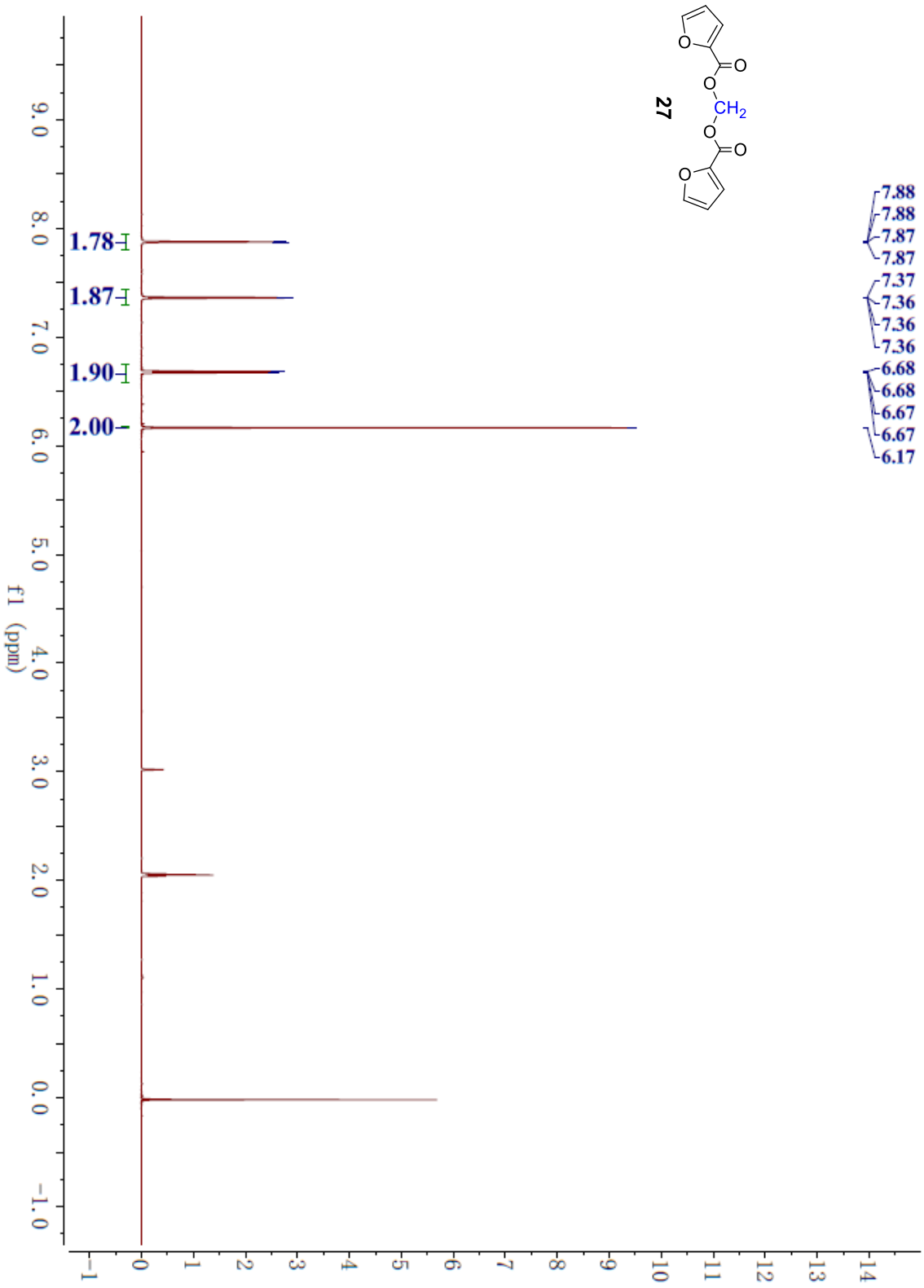

CARBON_01
205.99
165.05
146.43
134.15
130.75
128.92
128.38
116.83
79.36
29.38
29.26
29.13
29.00
28.87
28.74
28.61
23
O
O
CH2
O
O
f1 (ppm)

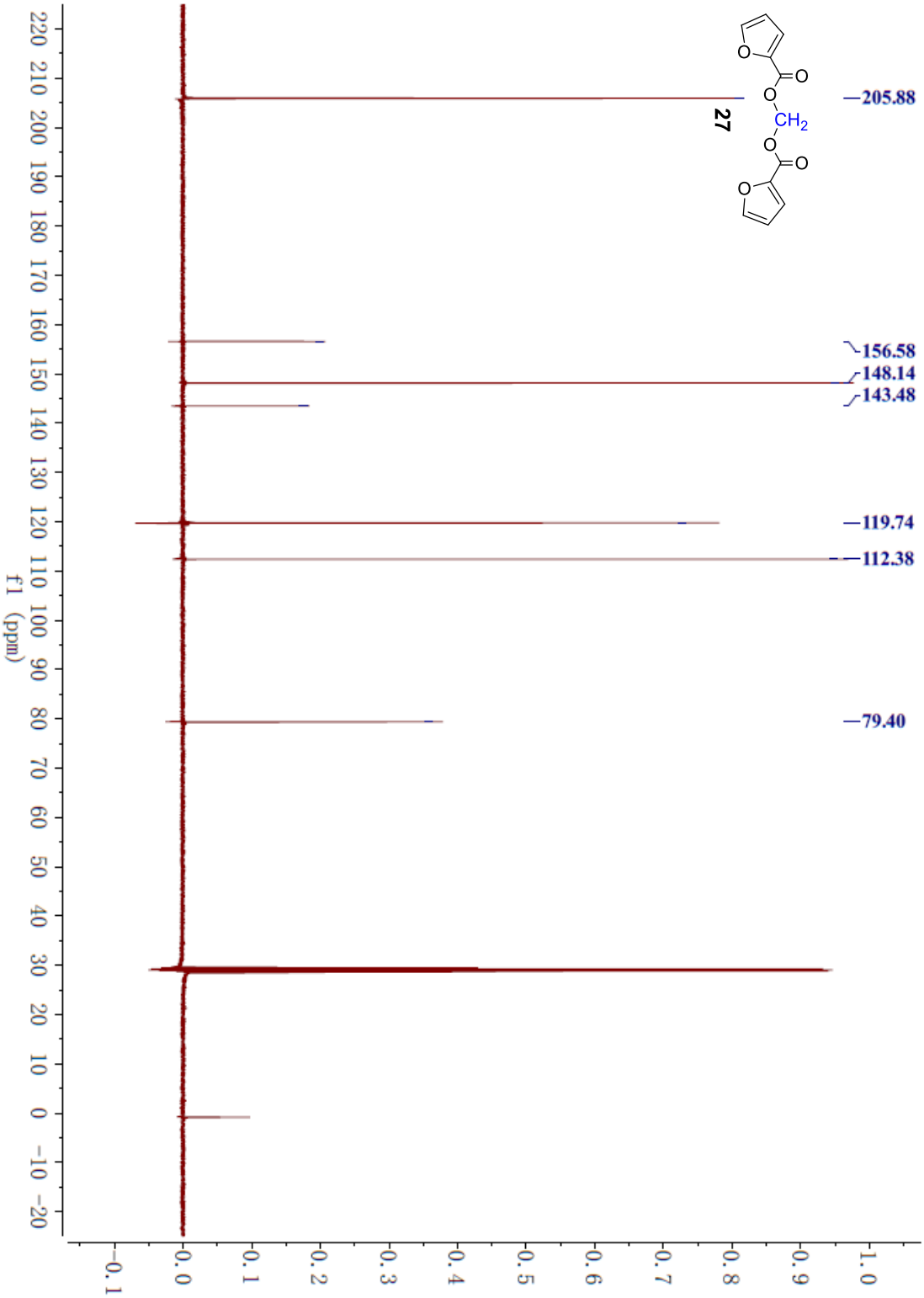

PROTON_01
24
f1 (ppm)
1.81
3.70
5.96
2.00
4.00
4.48
7.73
7.73
7.70
7.70
7.67
7.67
7.67
7.66
7.66
7.42
7.42
7.41
6.58
6.56

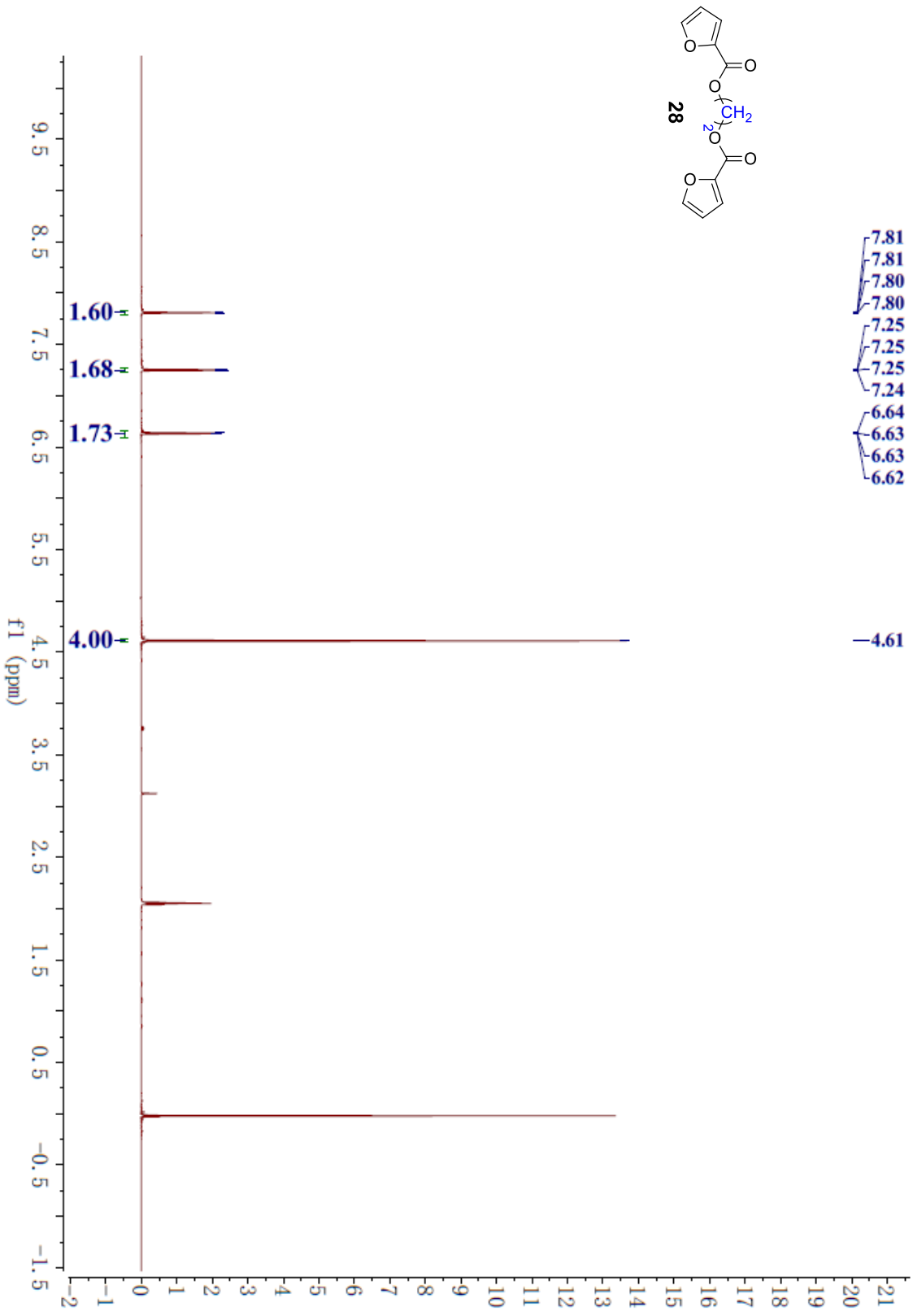

CARBON_01
205.97
166.22
145.02
134.29
130.46
128.93
128.16
117.70
62.22
29.38
29.25
29.12
28.99
28.86
28.74
28.61
24
f1 (ppm)

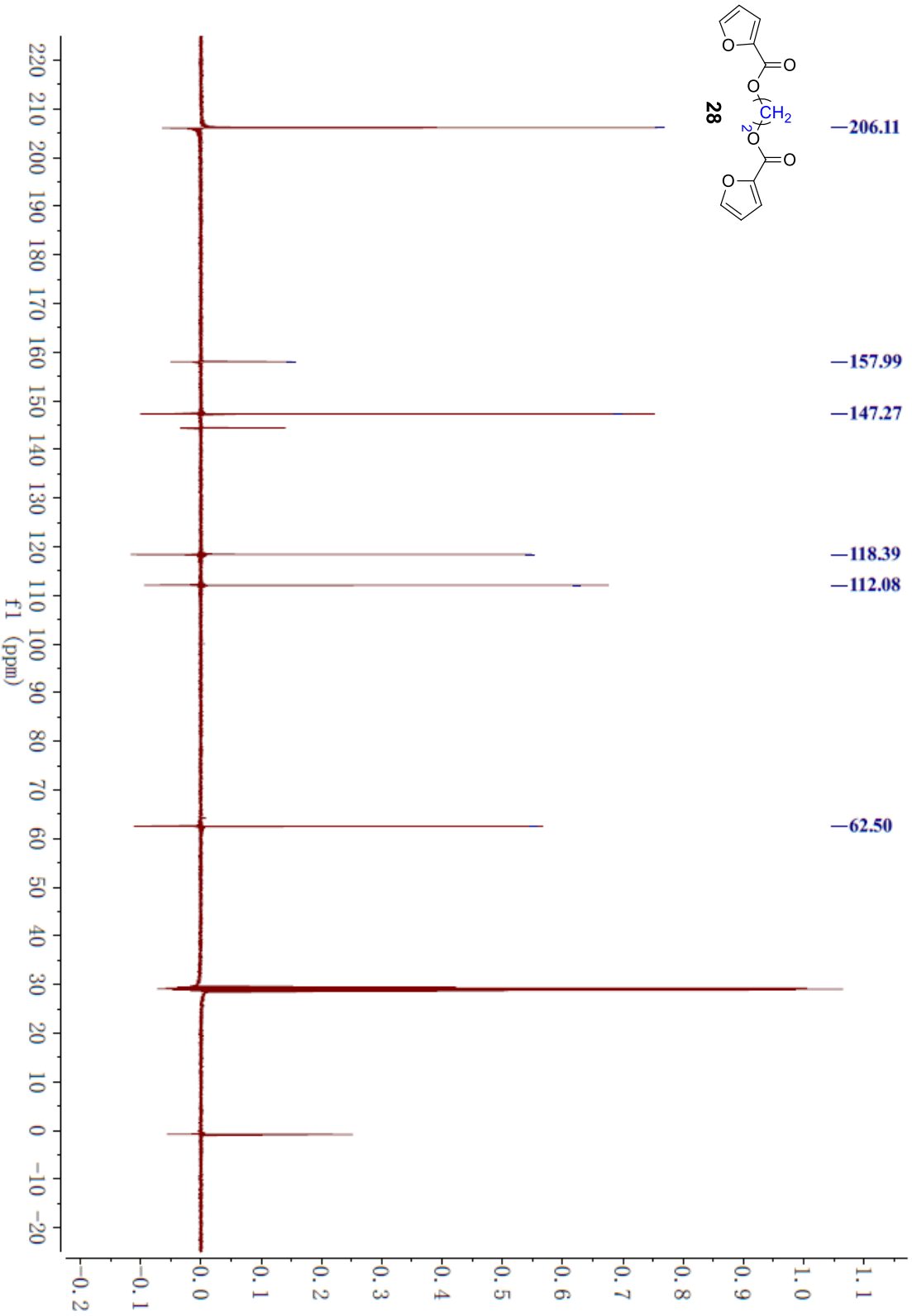

25
O
O
CH2
3
O
O
7.72
7.68
7.67
7.66
7.65
7.65
7.42
7.41
7.39
6.58
6.54
4.35
4.34
4.32
2.15
2.14
2.12
2.10
2.09
5.96
5.79
1.97
4.14
2.00
f1 (ppm)

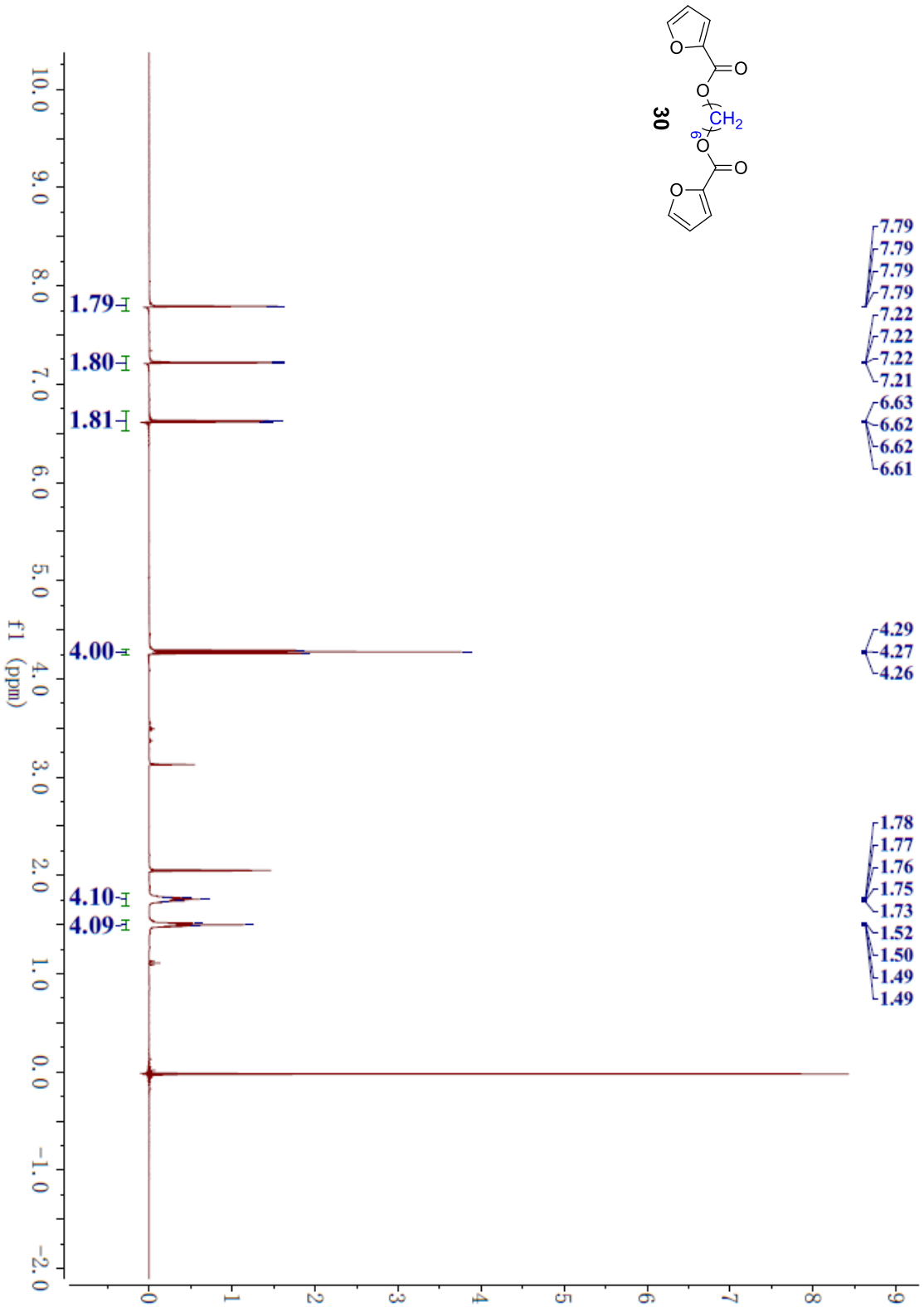

25
O
O
CH2
O
O
205.87
166.30
144.64
134.51
130.39
128.99
128.23
118.11
61.28
f1 (ppm)

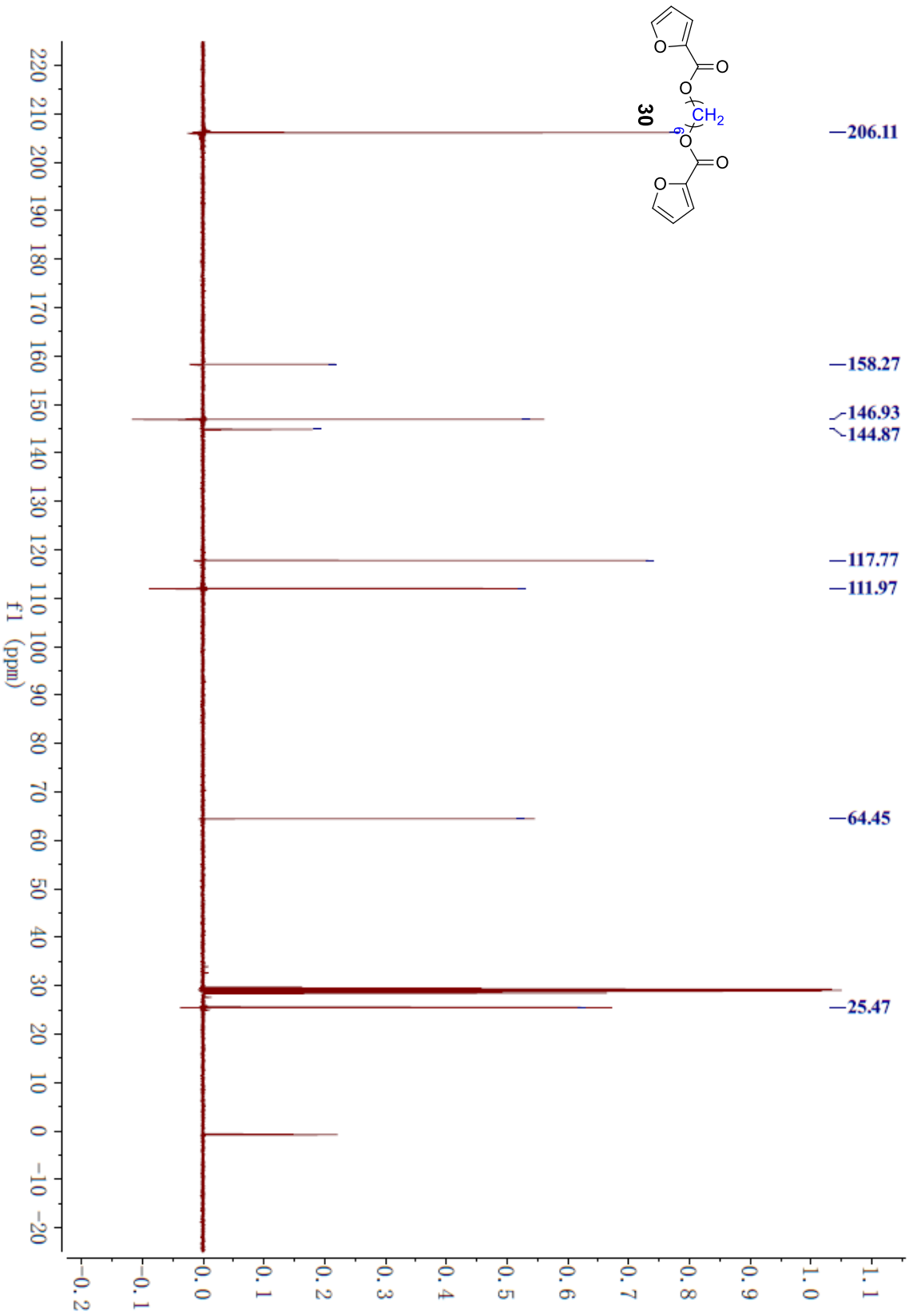

27
O
O
O
O
O
O
CH2
7.88
7.88
7.87
7.87
7.37
7.36
7.36
7.36
6.68
6.68
6.67
6.67
6.17
1.78
1.87
1.90
2.00
f1 (ppm)
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
−1.0
−1
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14

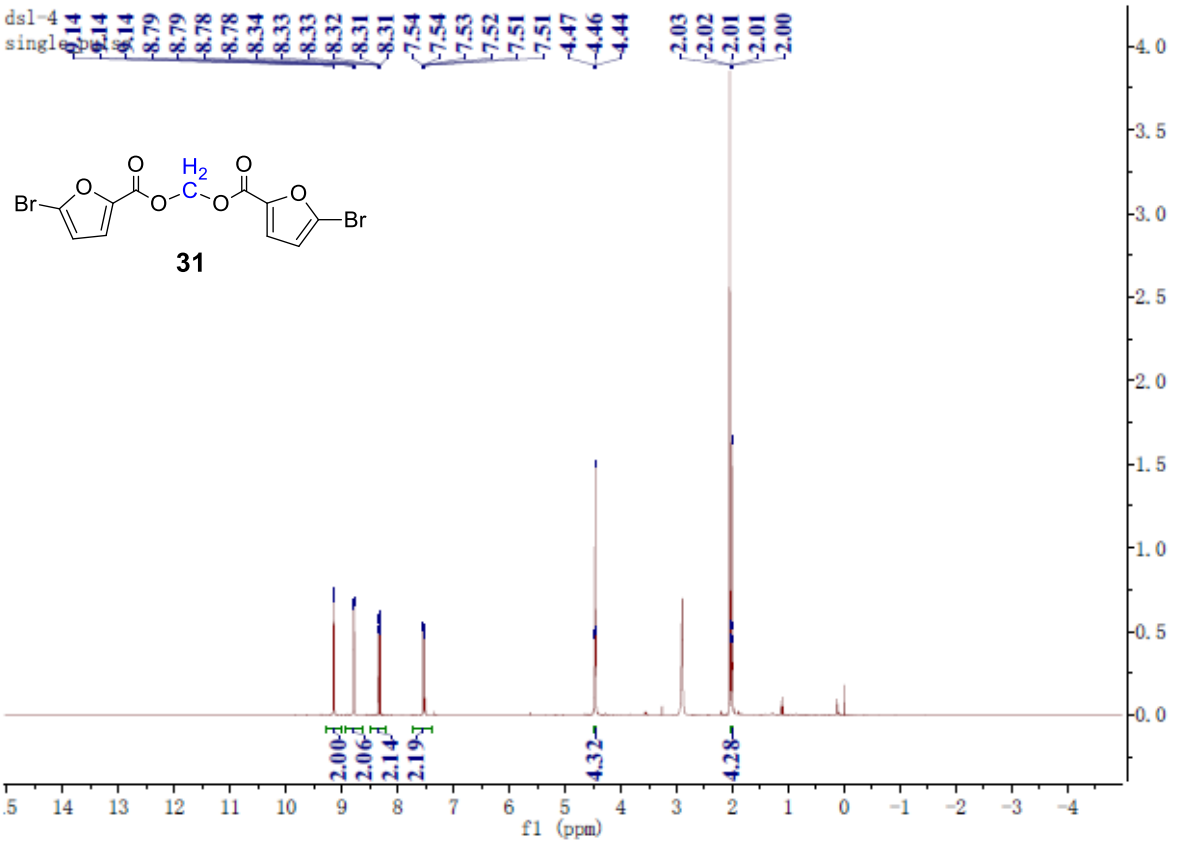

27
O
O
O
O
O
O
CH2
205.88
156.58
148.14
143.48
119.74
112.38
79.40
f1 (ppm)

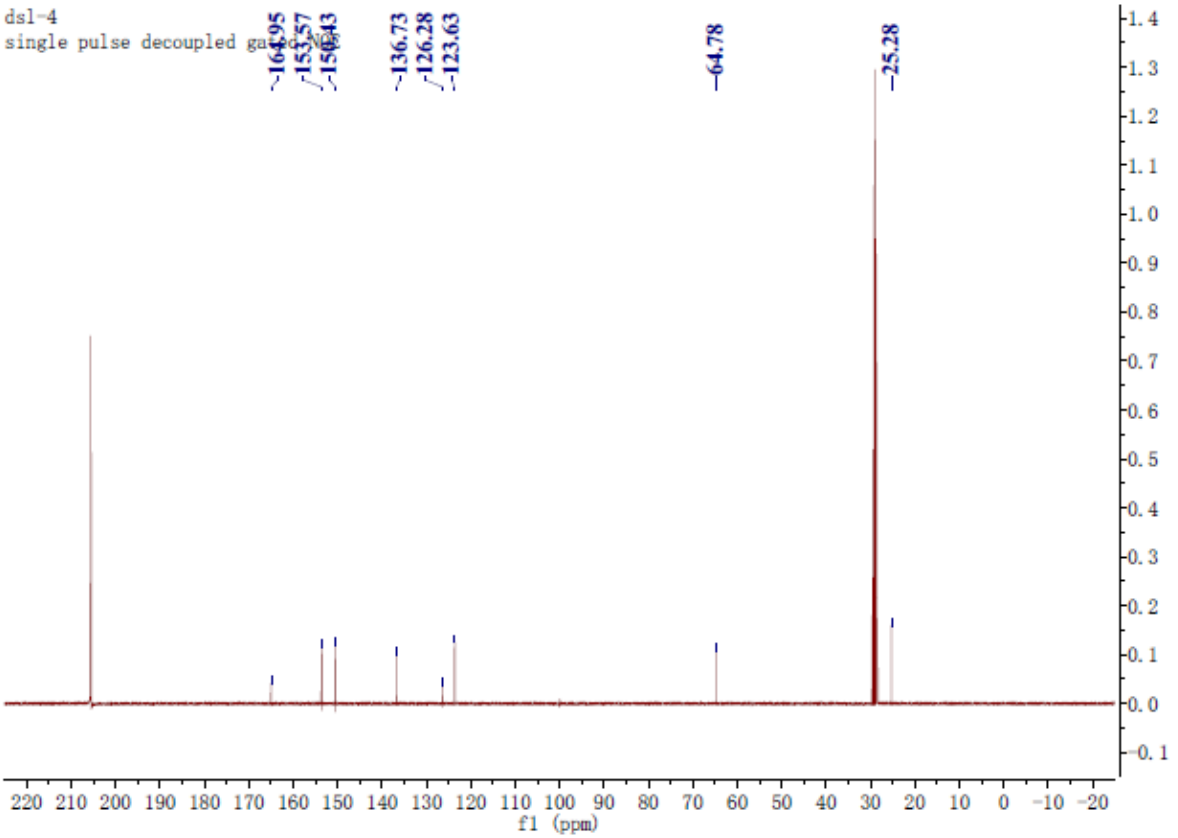

28
O
O
O
O
O
O
CH2
N
7.81
7.81
7.80
7.80
7.25
7.25
7.25
7.24
6.64
6.63
6.63
6.62
4.61
1.60
1.68
1.73
4.00
f1 (ppm)


28
206.11
157.99
147.27
118.39
112.08
62.50
f1 (ppm)

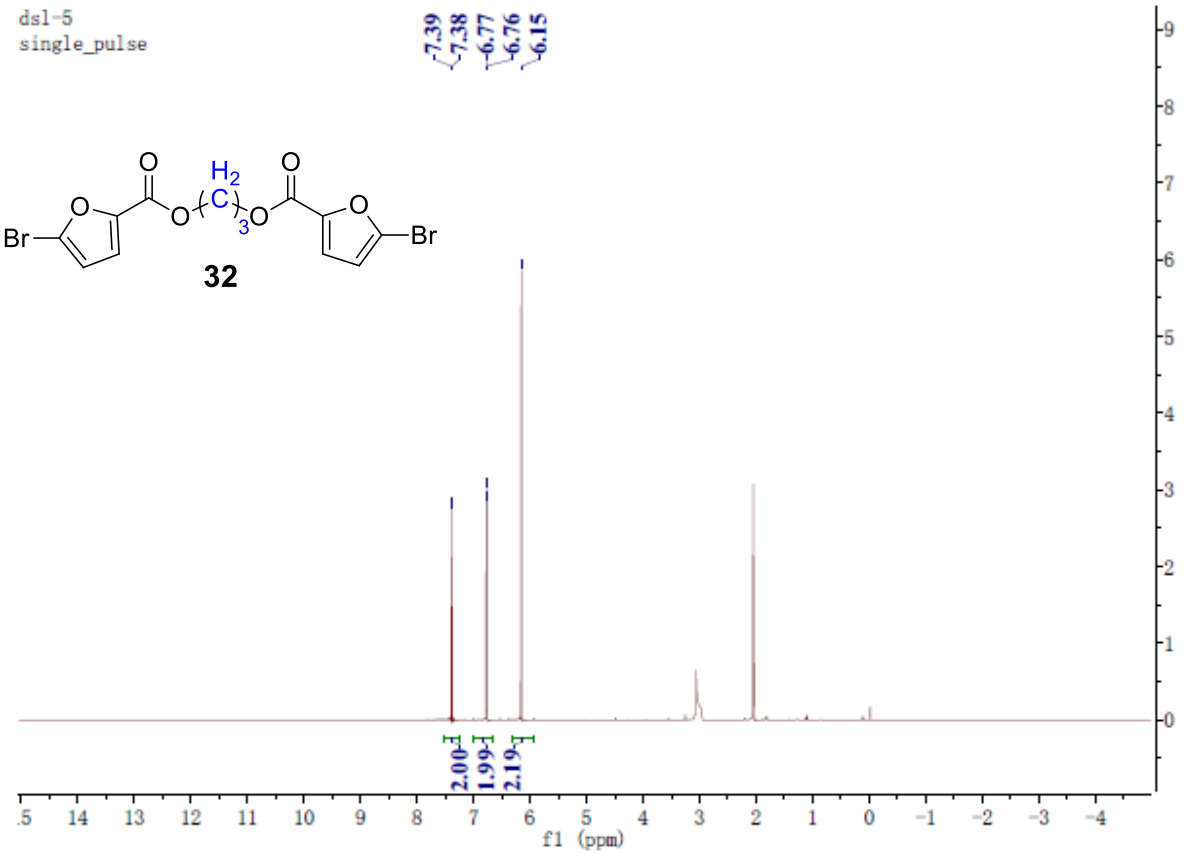

30
O
O
CH2
6
O
O
O
O
1.79
1.80
1.81
4.00
4.10
4.09
7.79
7.79
7.79
7.79
7.22
7.22
7.22
7.21
6.63
6.62
6.62
6.61
4.29
4.27
4.26
1.78
1.77
1.76
1.75
1.73
1.52
1.50
1.49
1.49
f1 (ppm)

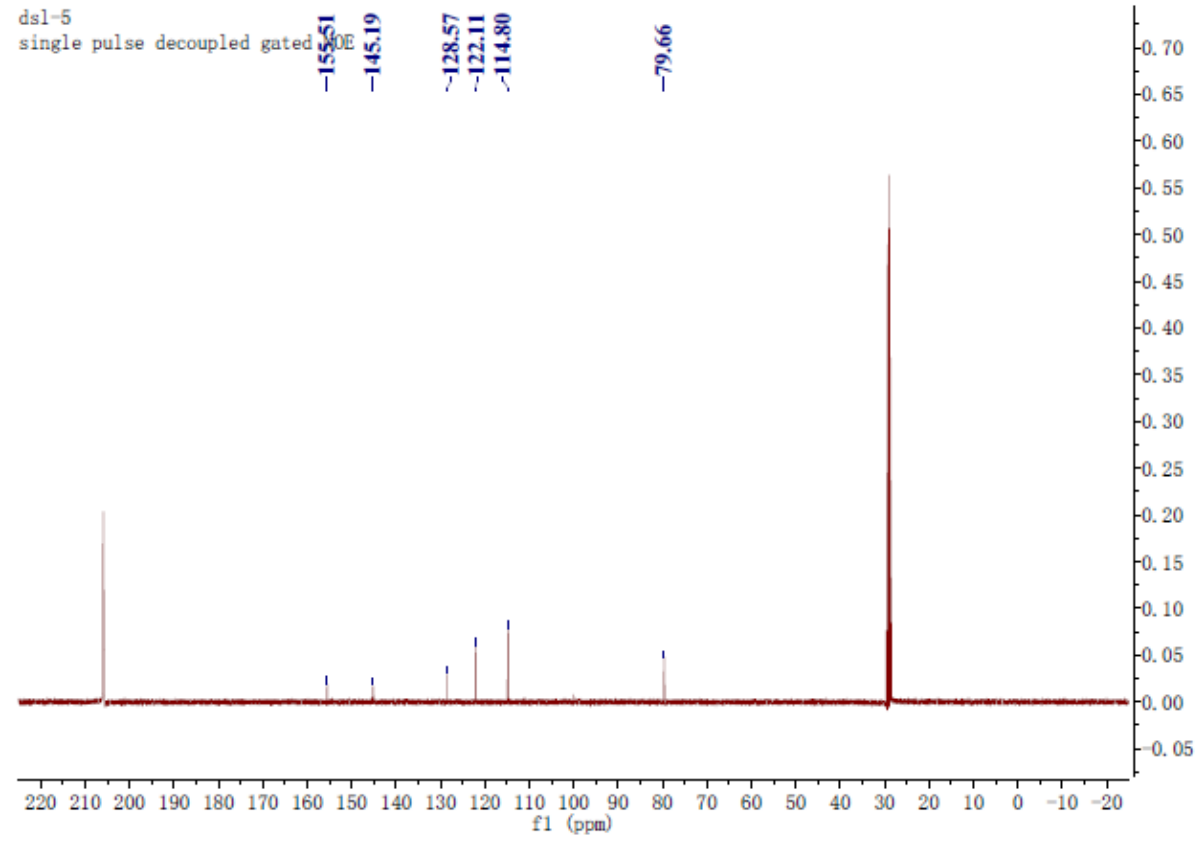

206.11
158.27
146.93
144.87
117.77
111.97
64.45
25.47
30
CH2
9
f1 (ppm)



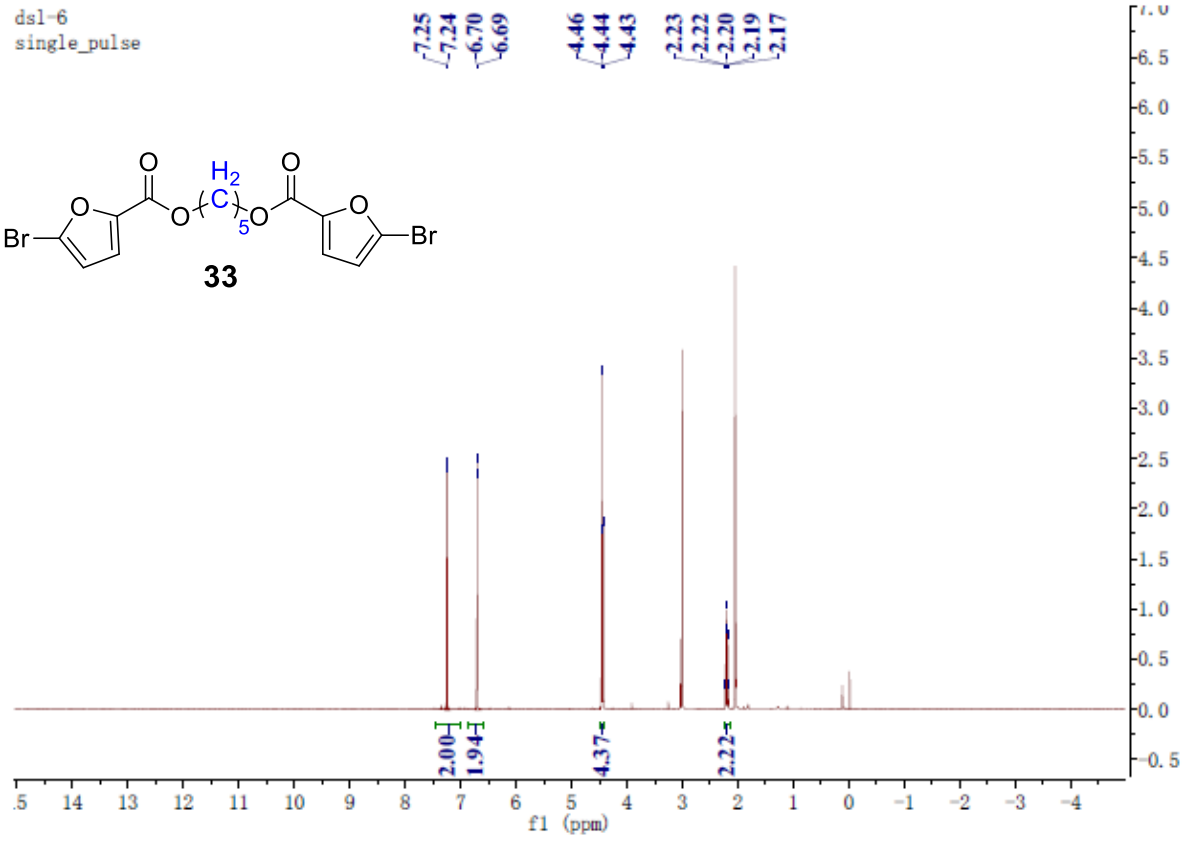


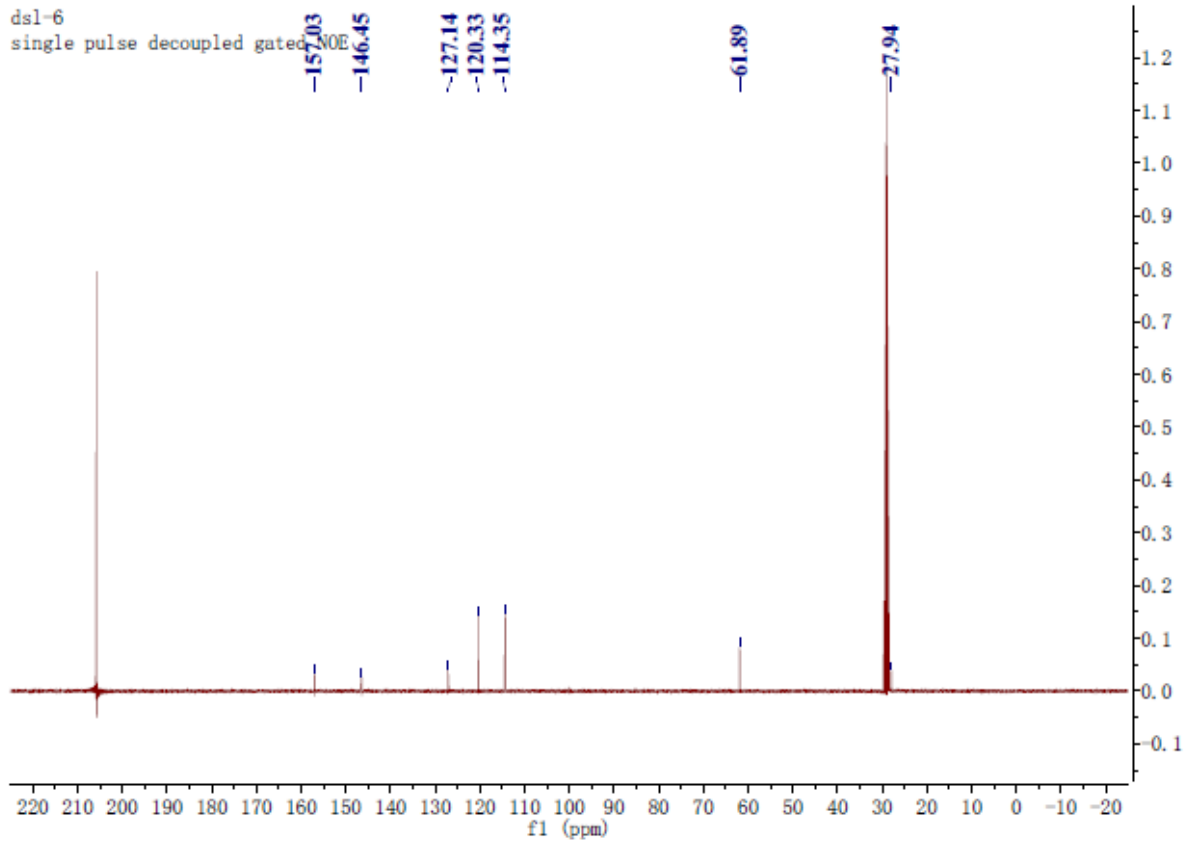
^{1}H and ^{13}C NMR Spectra of compound **31**



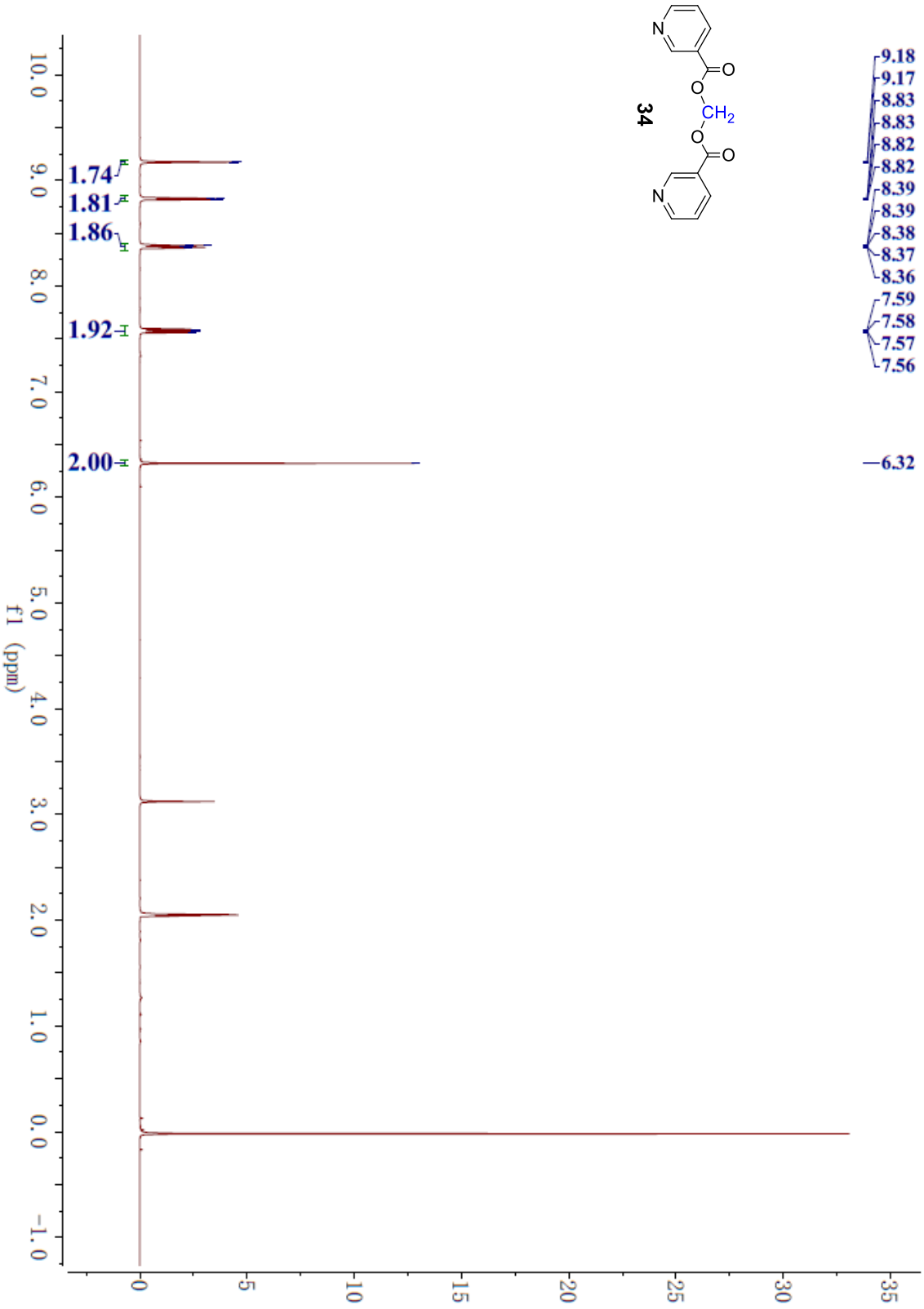


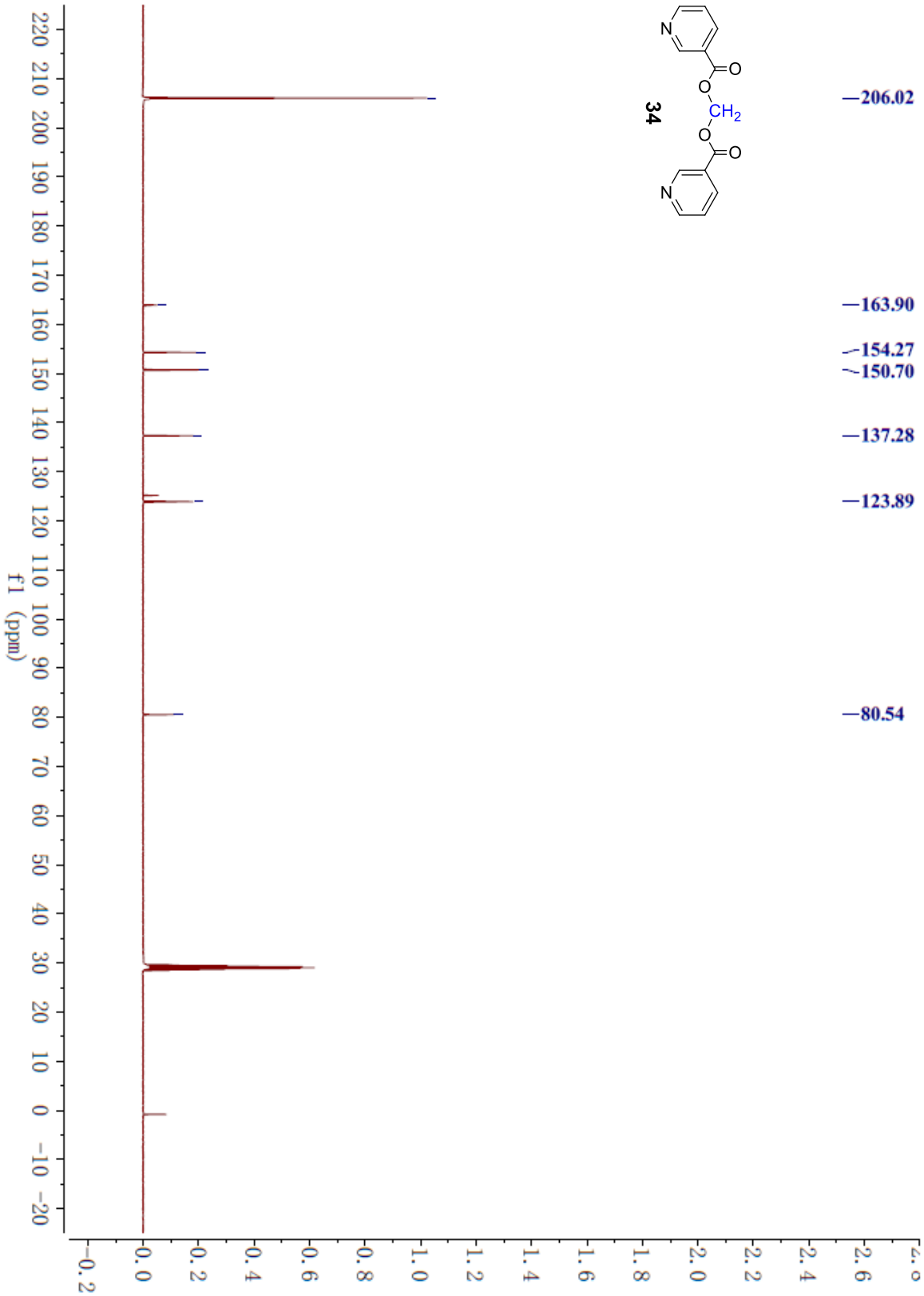
^{1}H and ^{13}C NMR Spectra of compound **32**

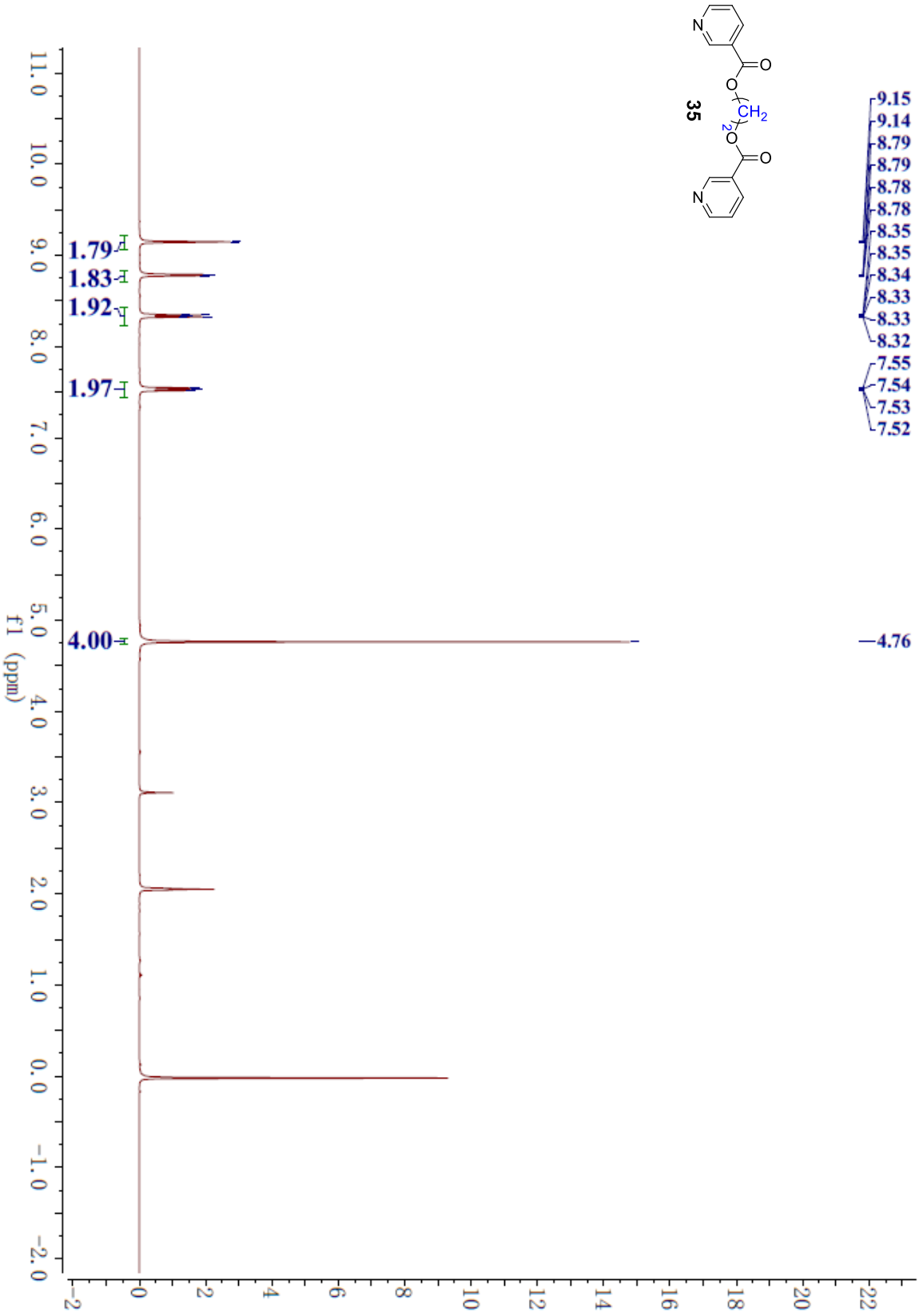


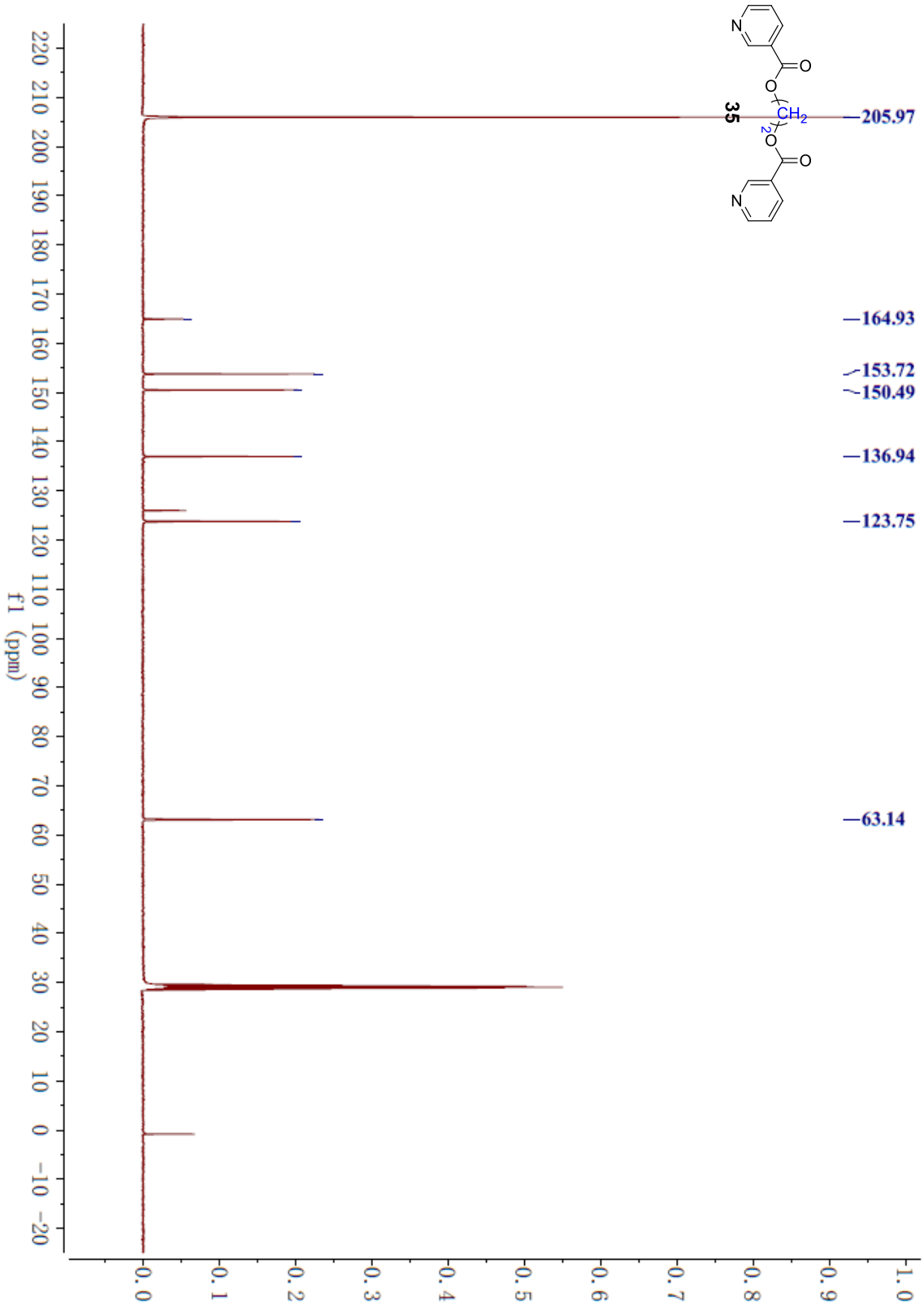


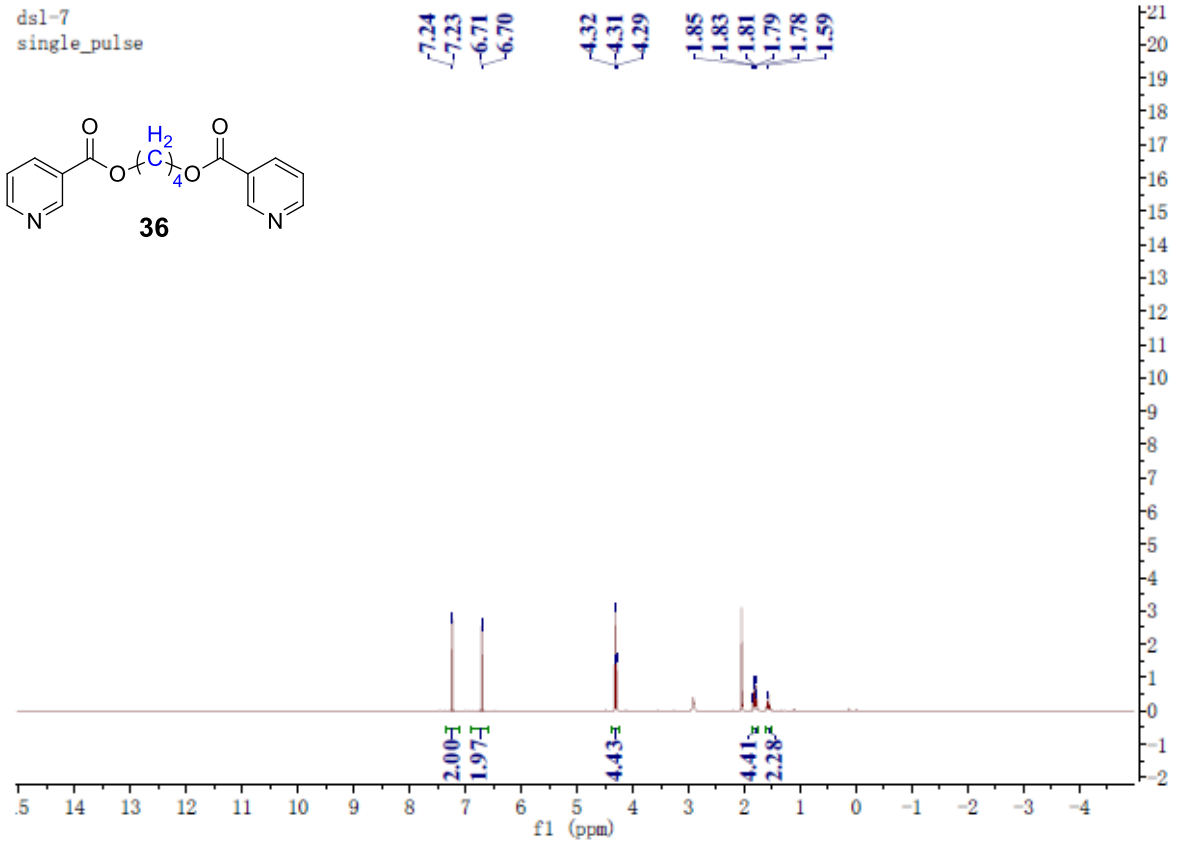
^{1}H and ^{13}C NMR Spectra of compound **33**

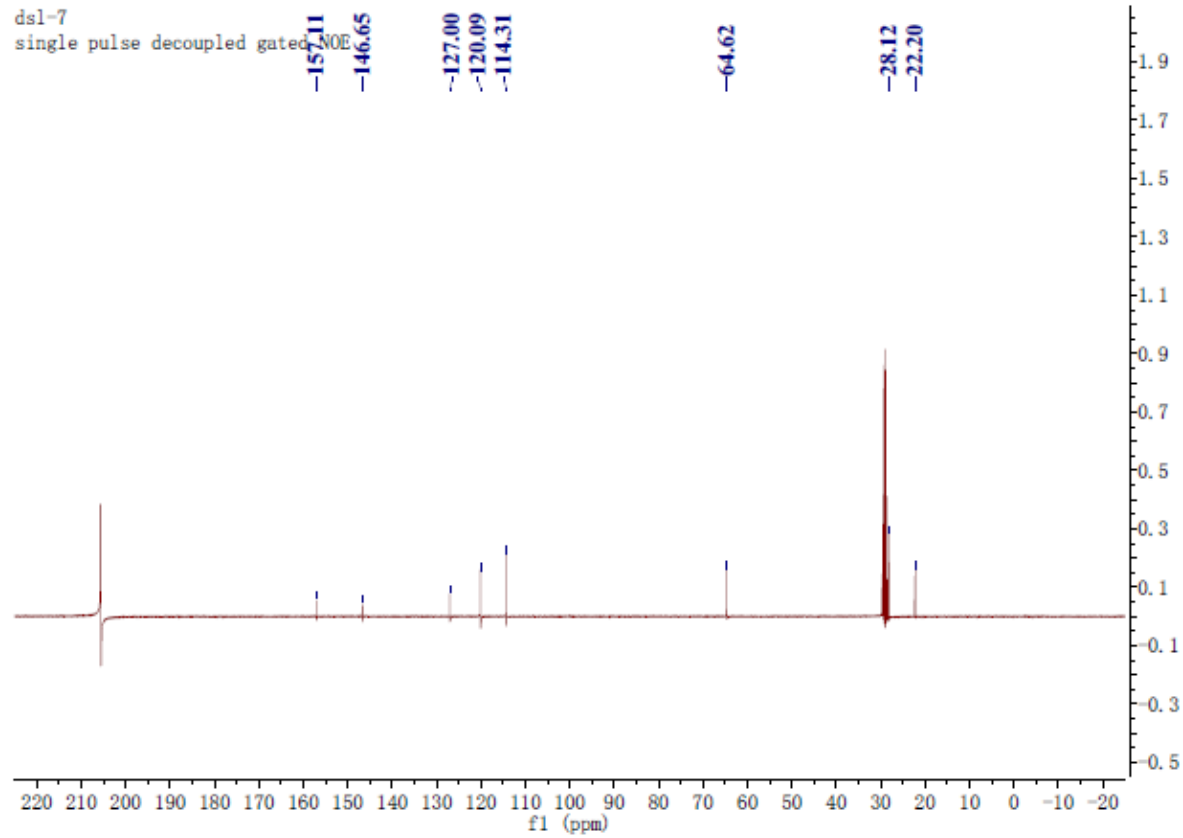

34
9.18
9.17
8.83
8.83
8.82
8.82
8.39
8.39
8.38
8.37
8.36
7.59
7.58
7.57
7.56
6.32
1.74
1.81
1.86
1.92
2.00
f1 (ppm)


34
206.02
163.90
154.27
150.70
137.28
123.89
80.54
f1 (ppm)


35
1.79
1.83
1.92
1.97
4.00
9.15
9.14
8.79
8.79
8.78
8.78
8.35
8.35
8.34
8.33
8.33
8.32
7.55
7.54
7.53
7.52
4.76
f1 (ppm)


205.97
164.93
153.72
150.49
136.94
123.75
63.14
f1 (ppm)

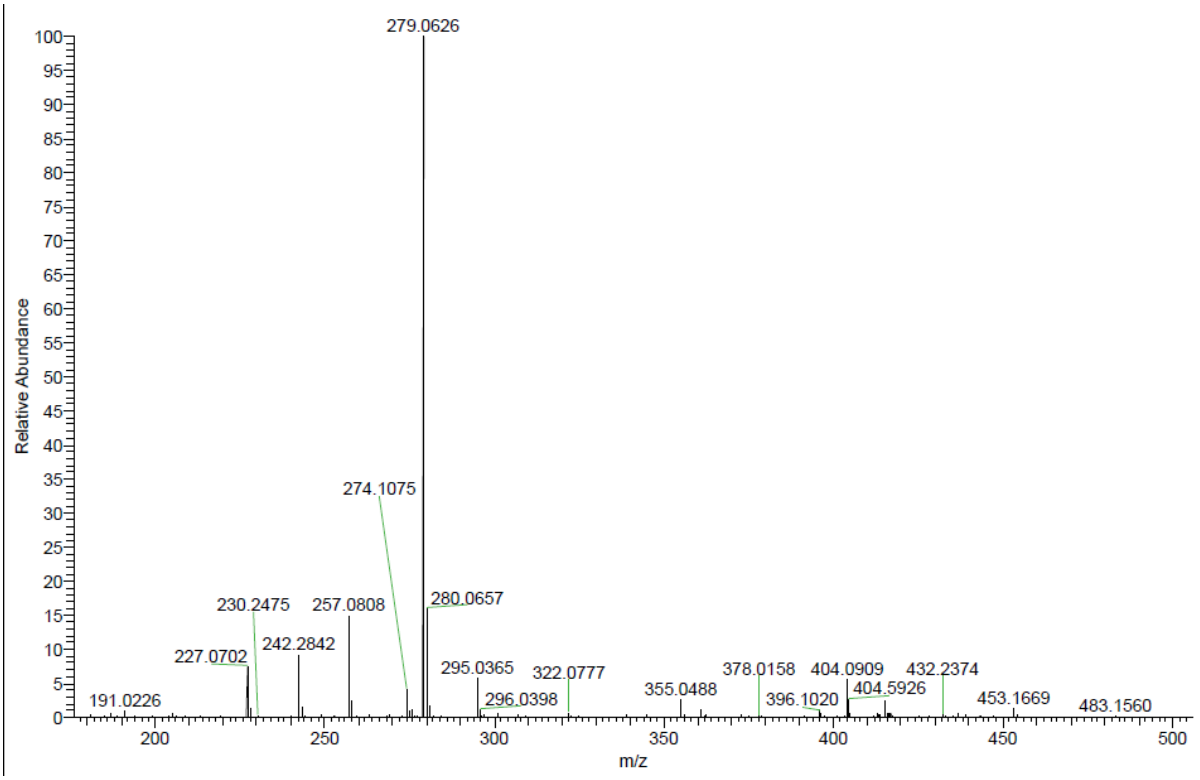




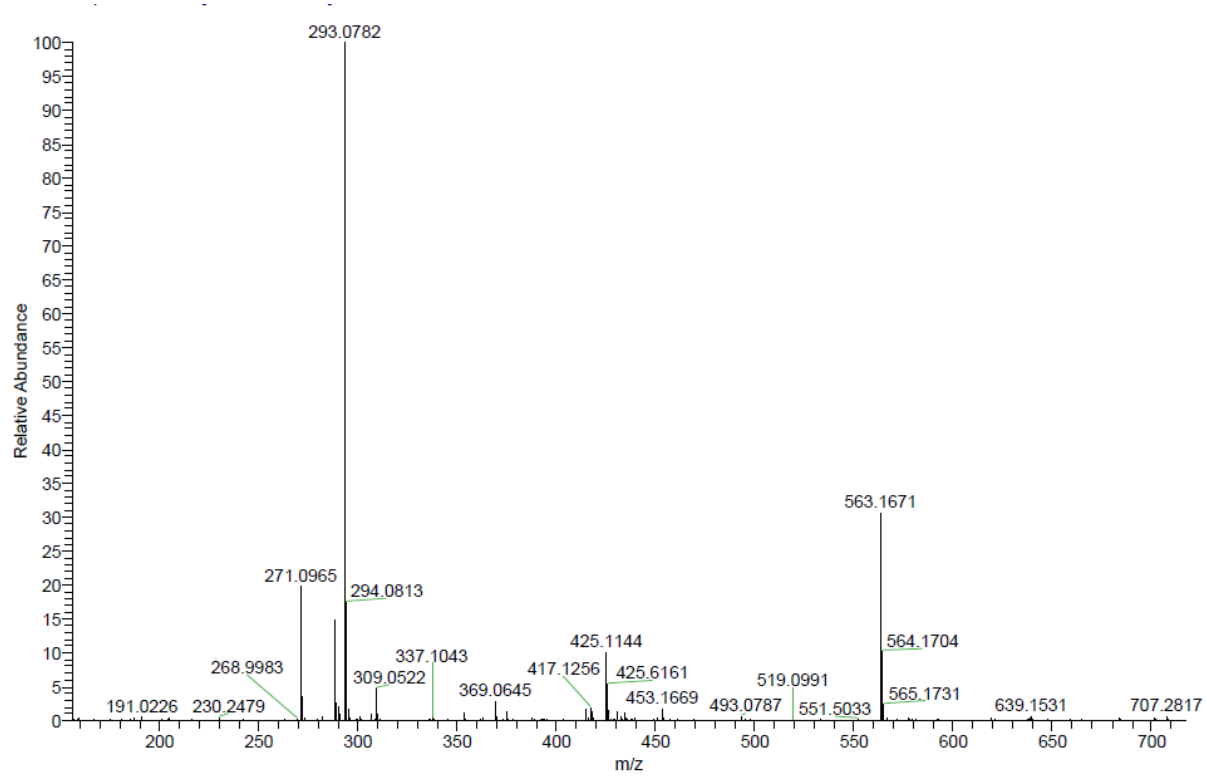
^{1}H and ^{13}C NMR Spectra of compound **36**

5. HRMS Spectra of the Products

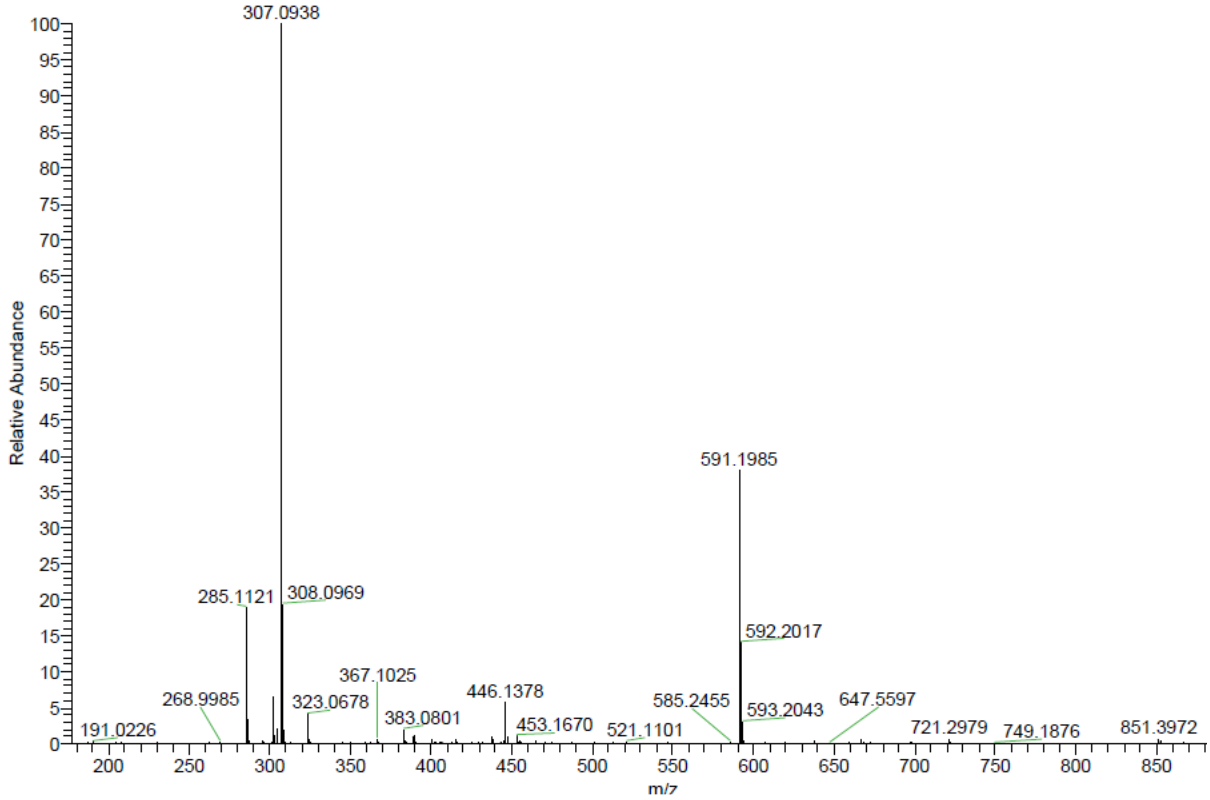
HRMS of **1**



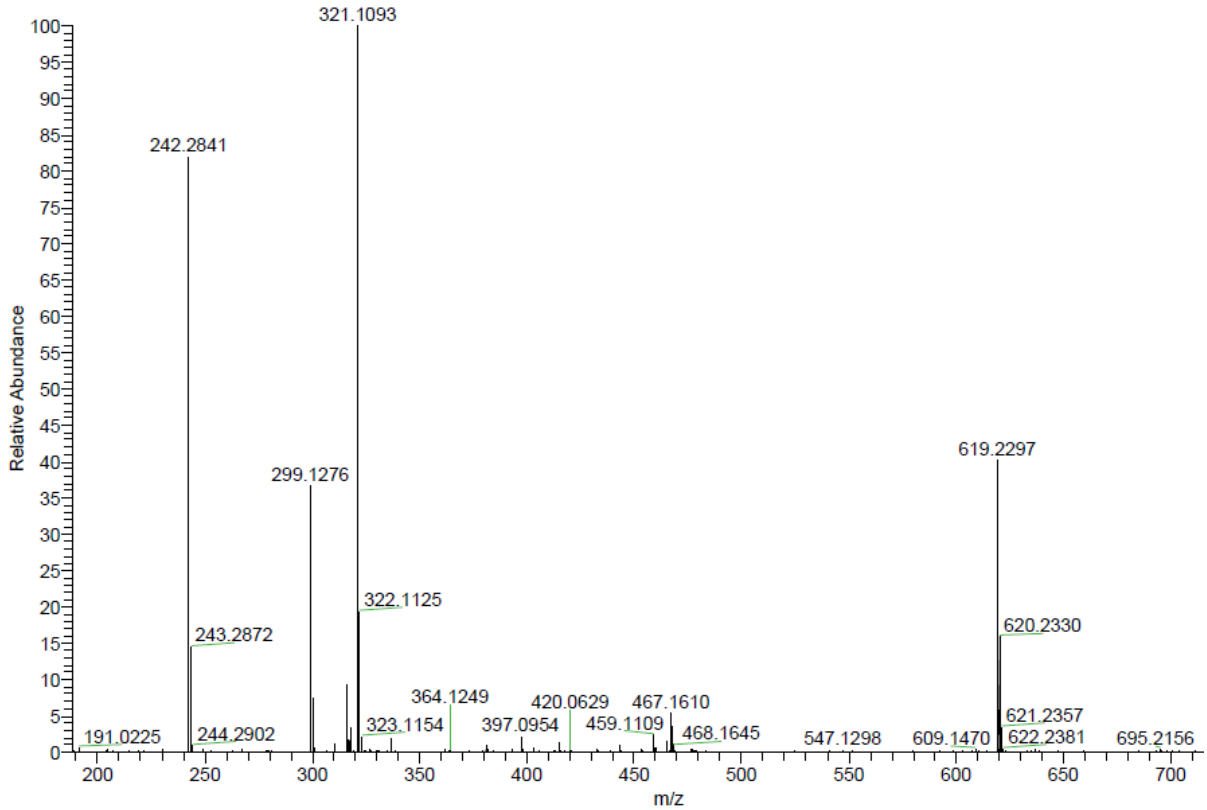
HRMS of **2**



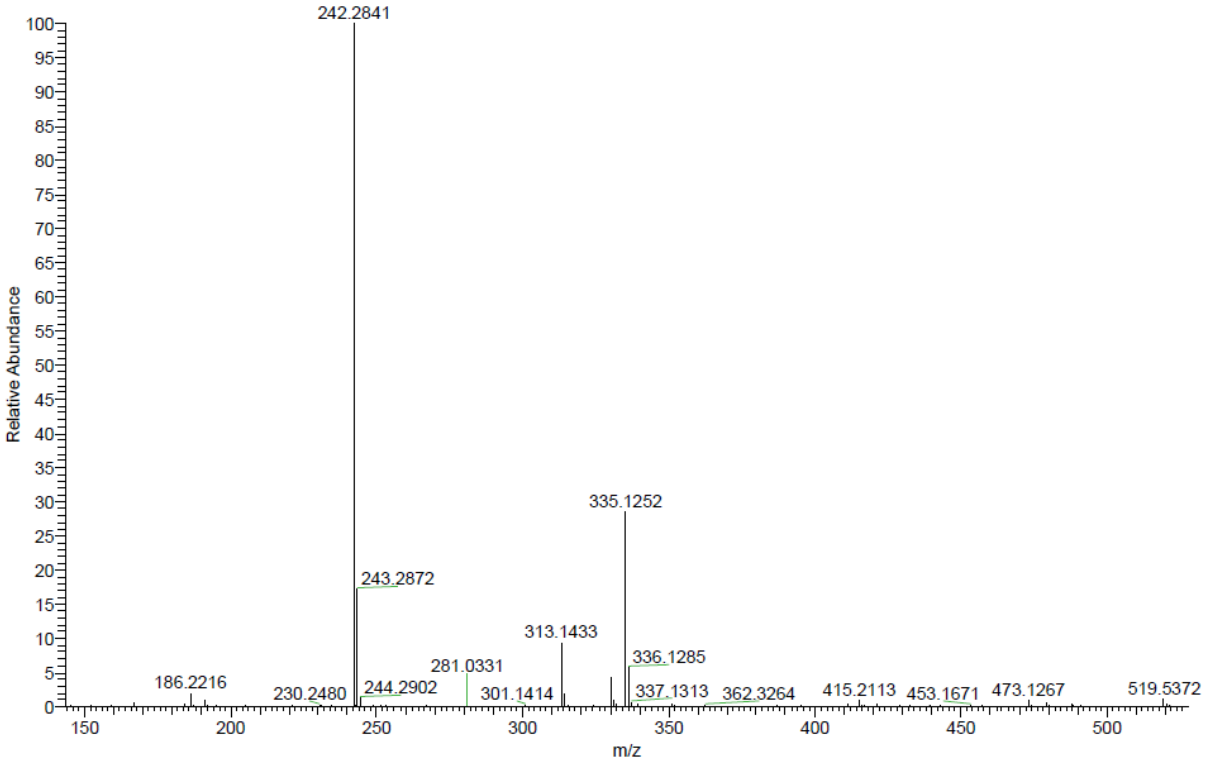
HRMS of **3**



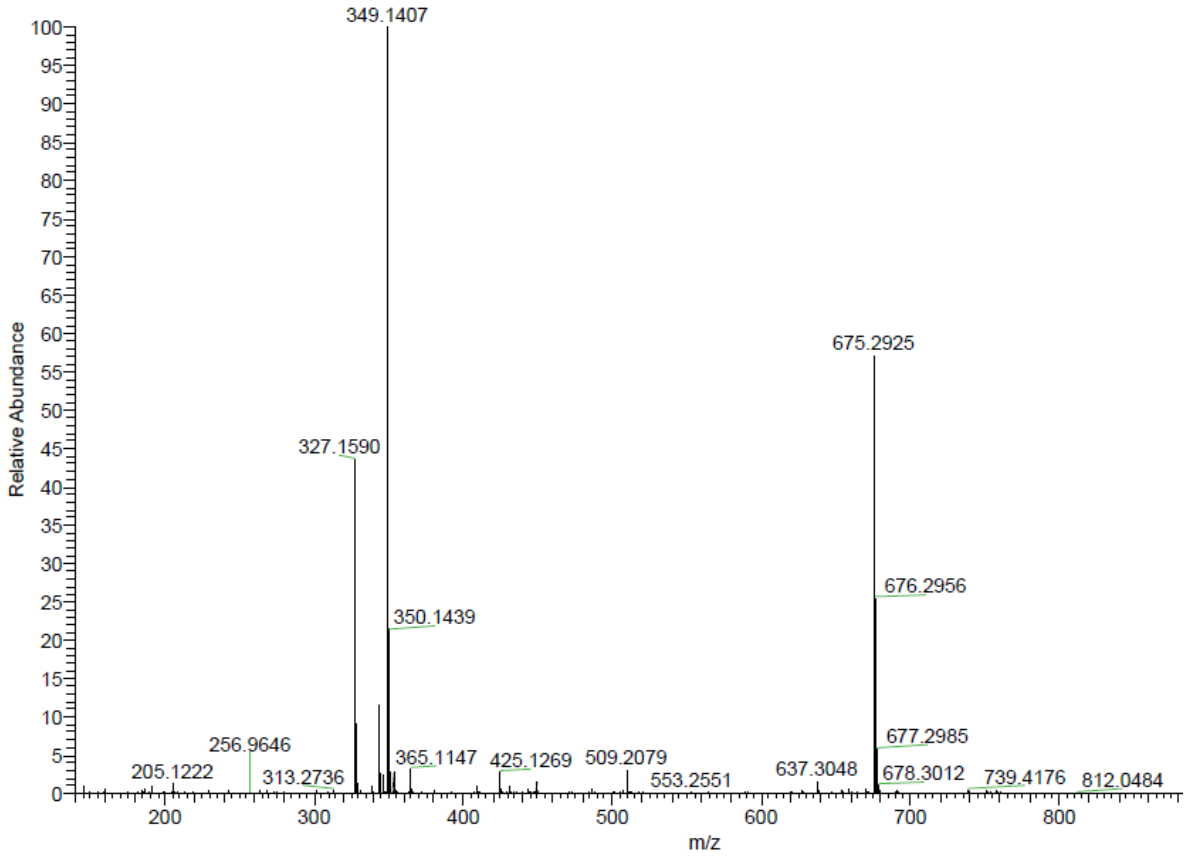
HRMS of **4**



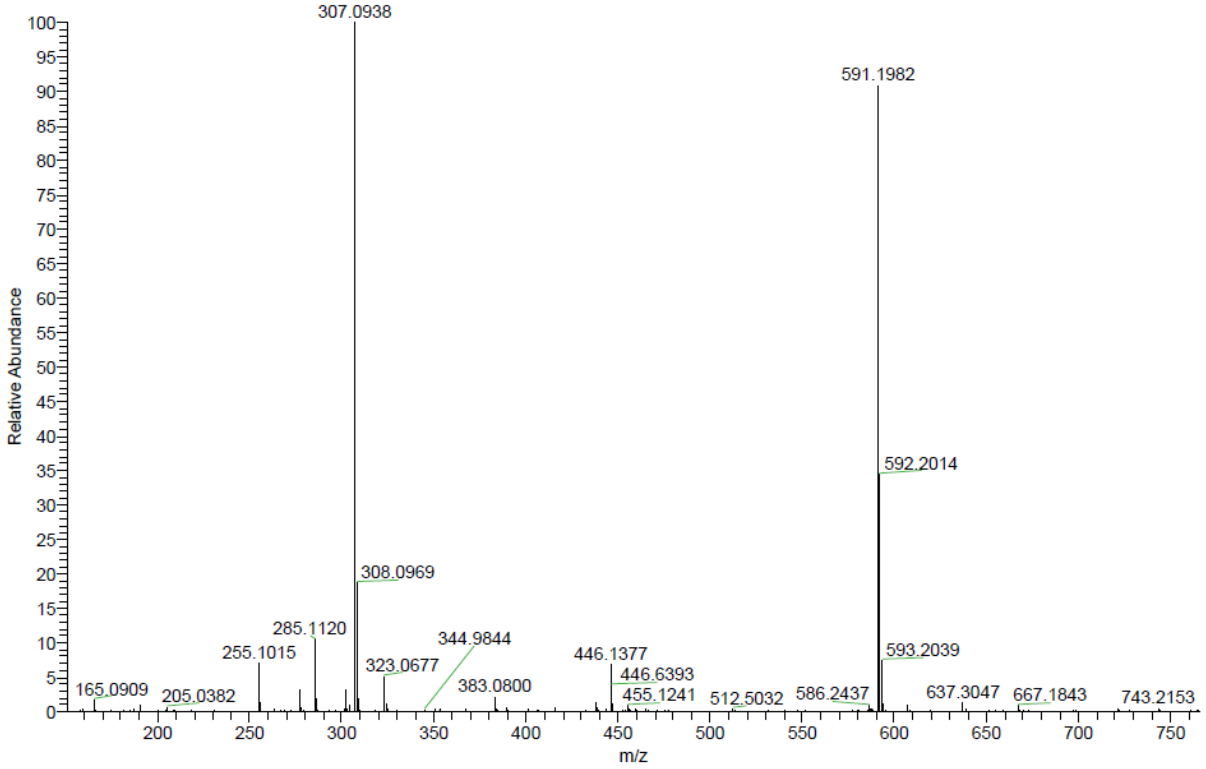
HRMS of **5**



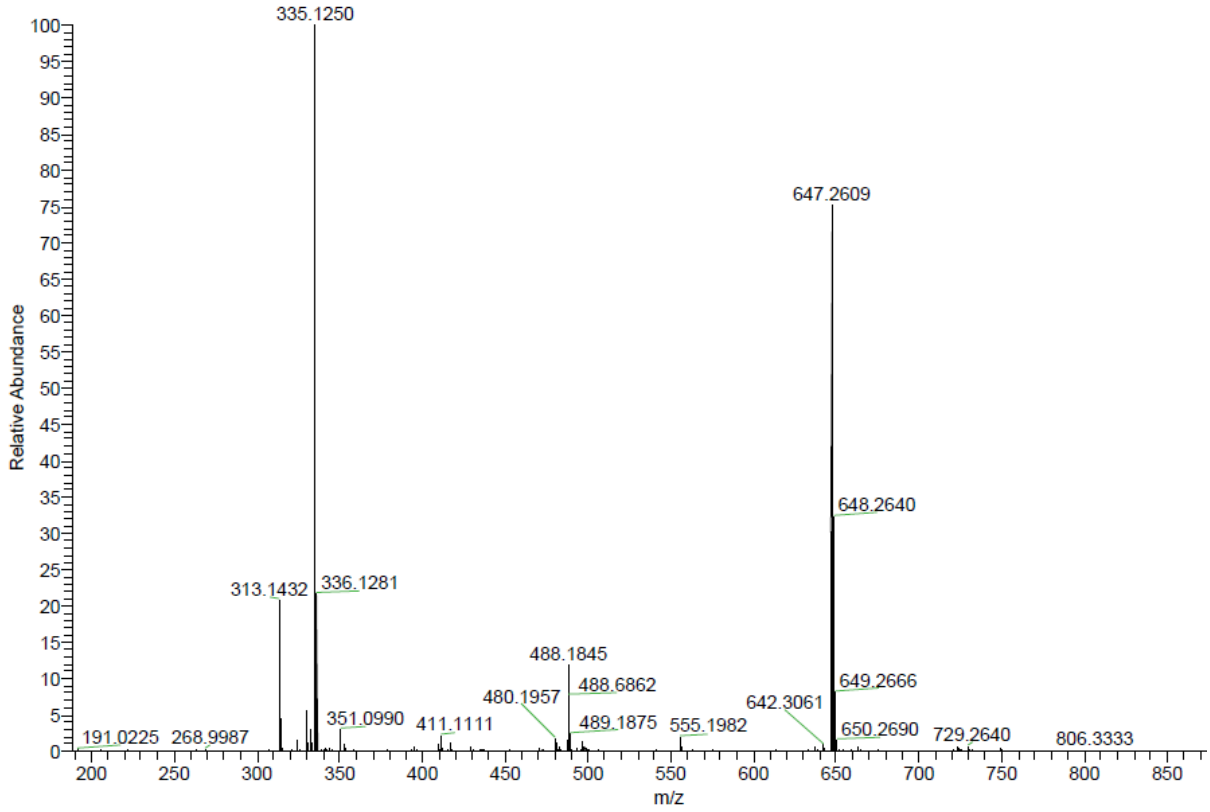
HRMS of **6**



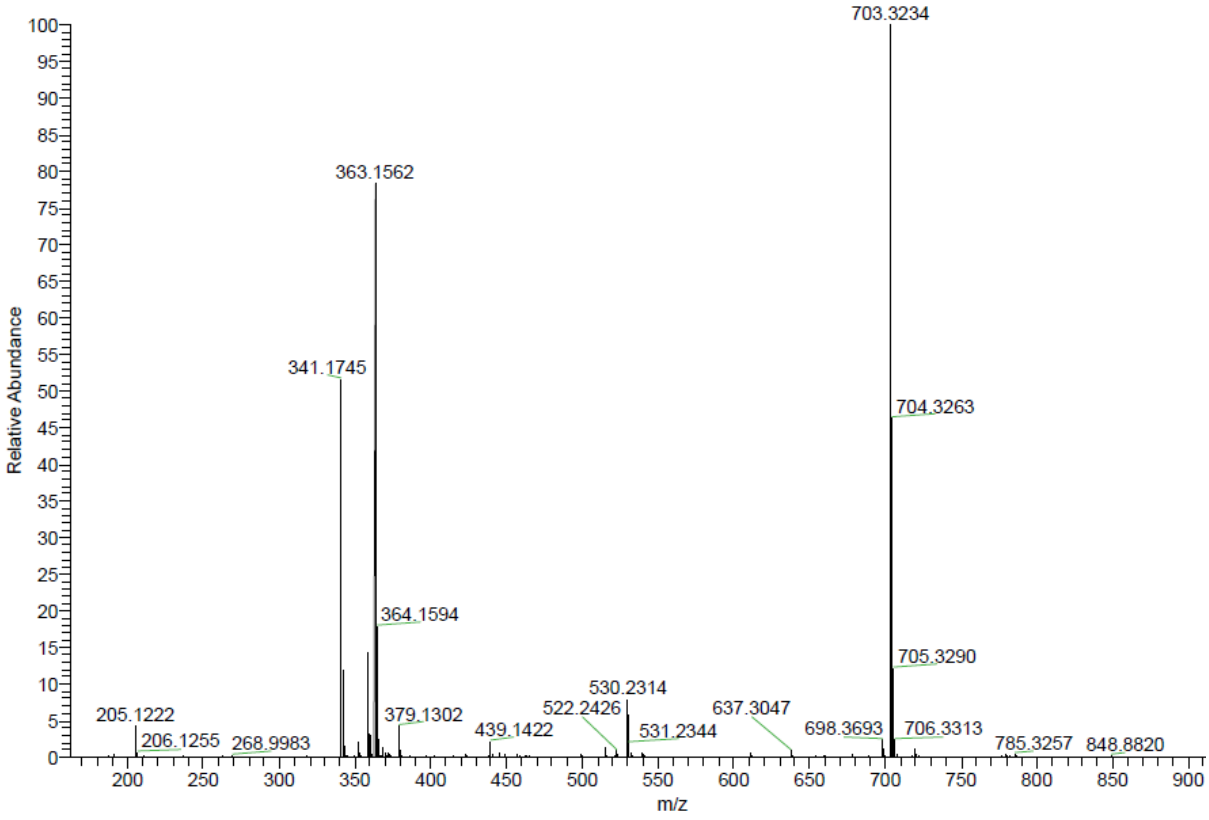
HRMS of **7**



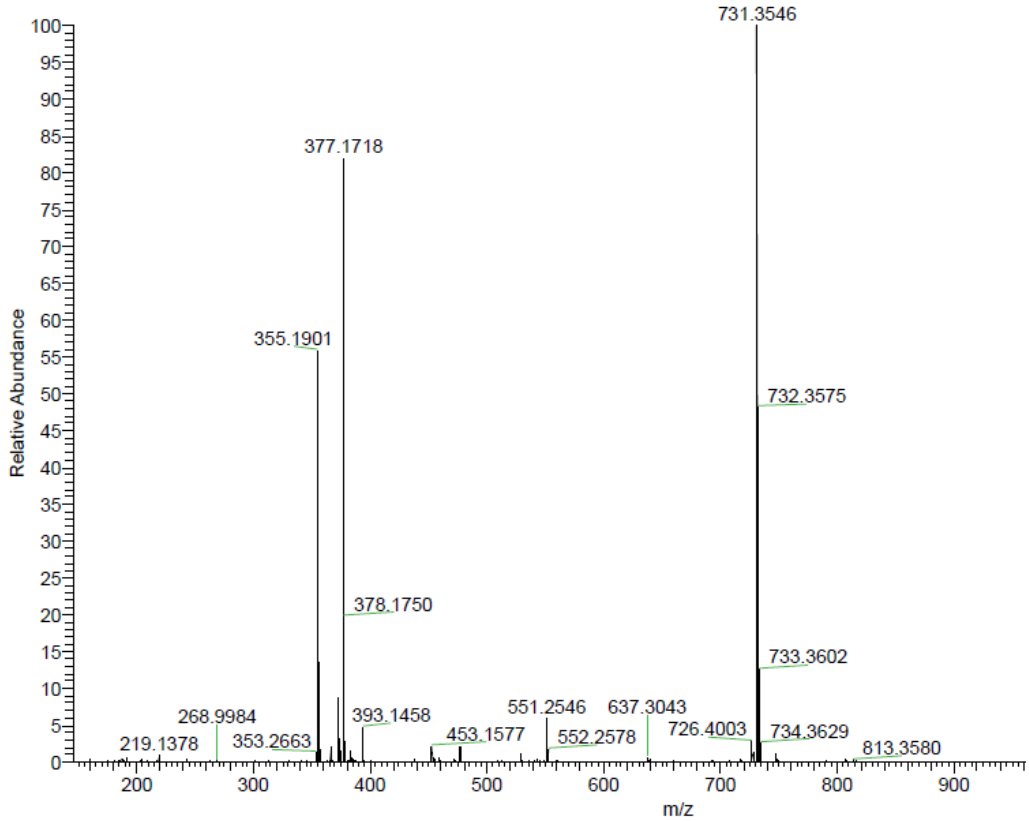
HRMS of **8**



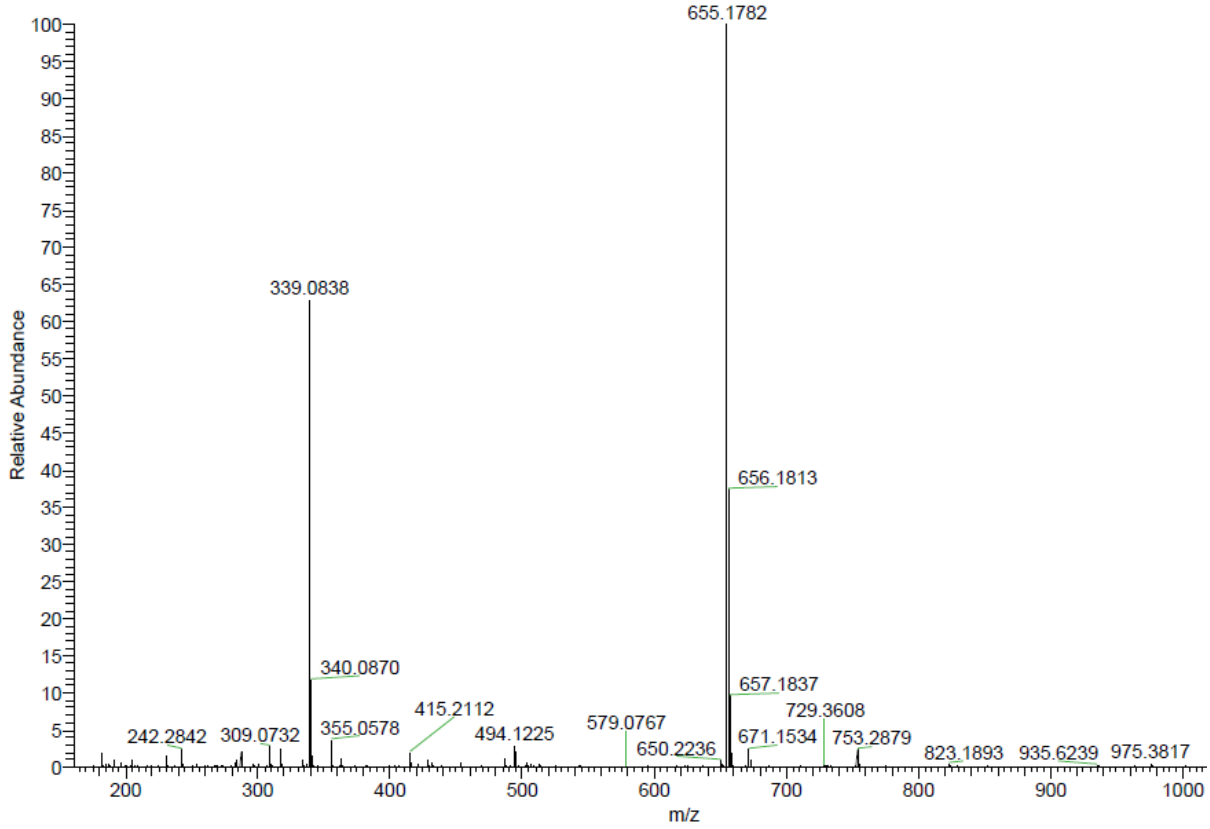
HRMS of **10**



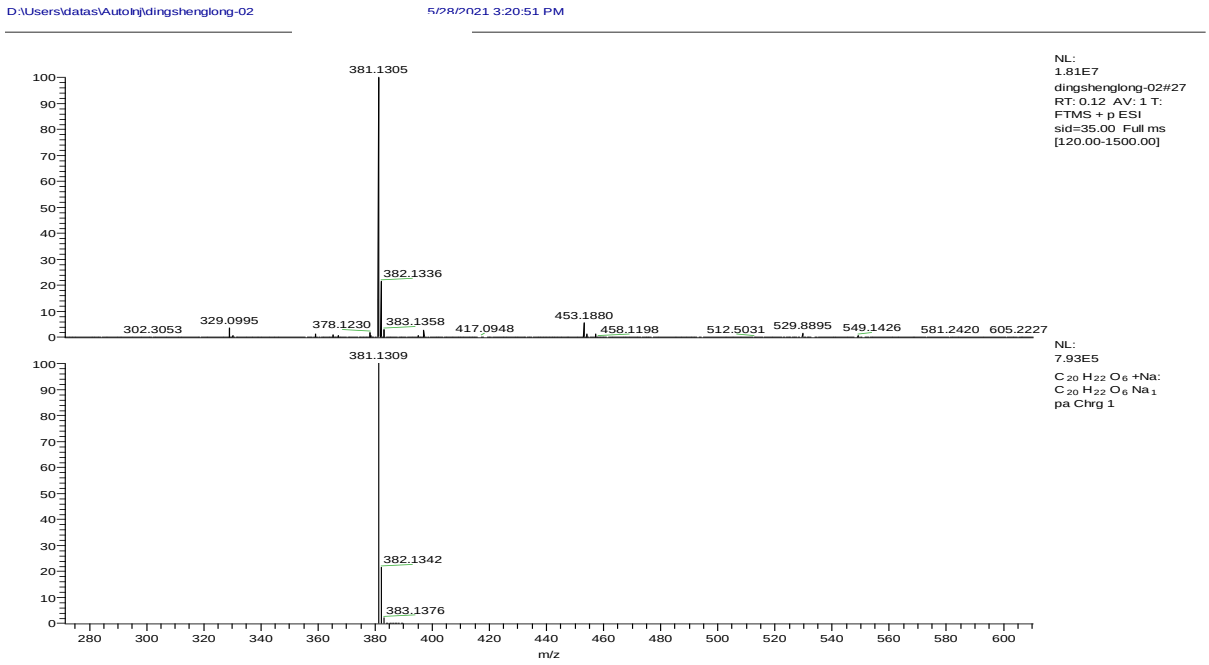
HRMS of **11**



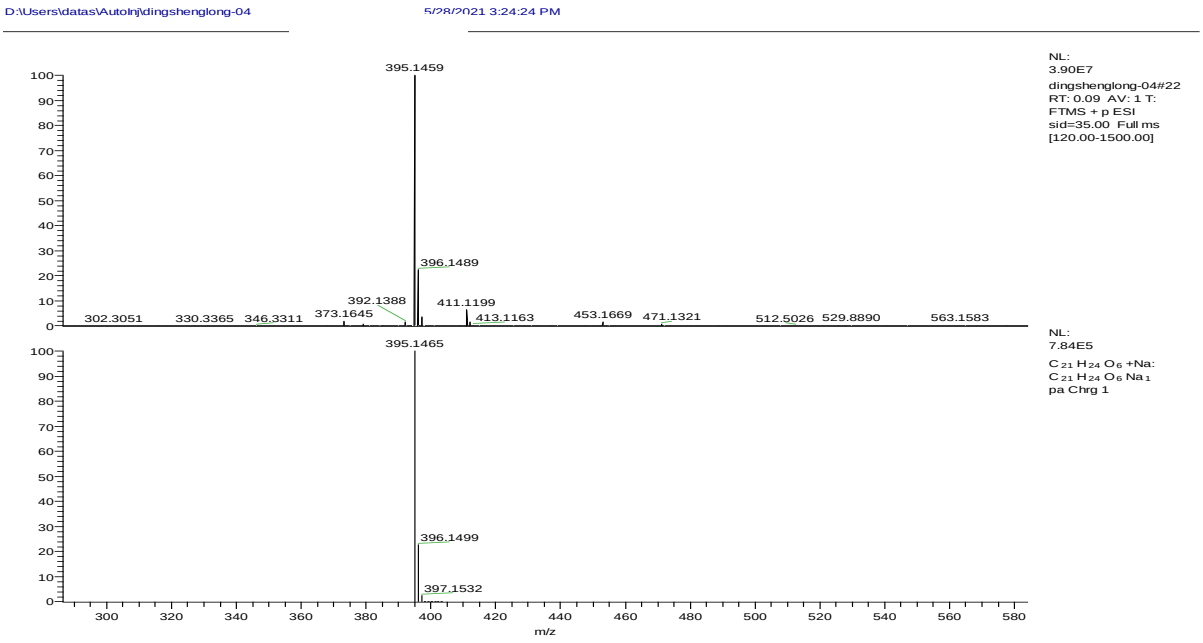
HRMS of **12**



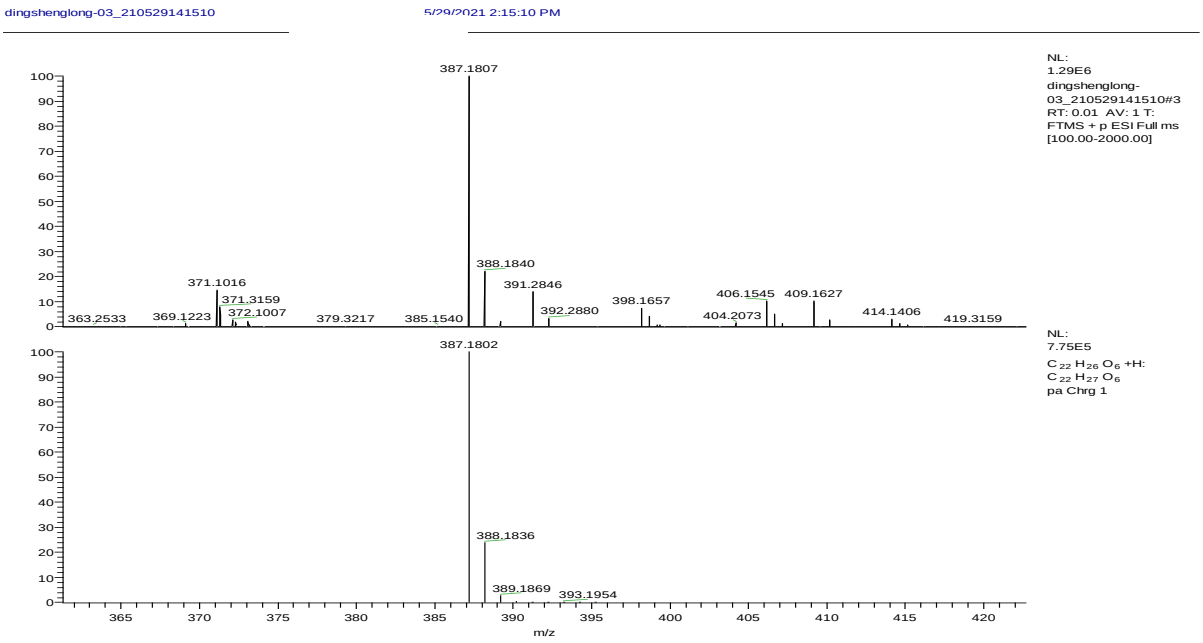
HRMS of **13**



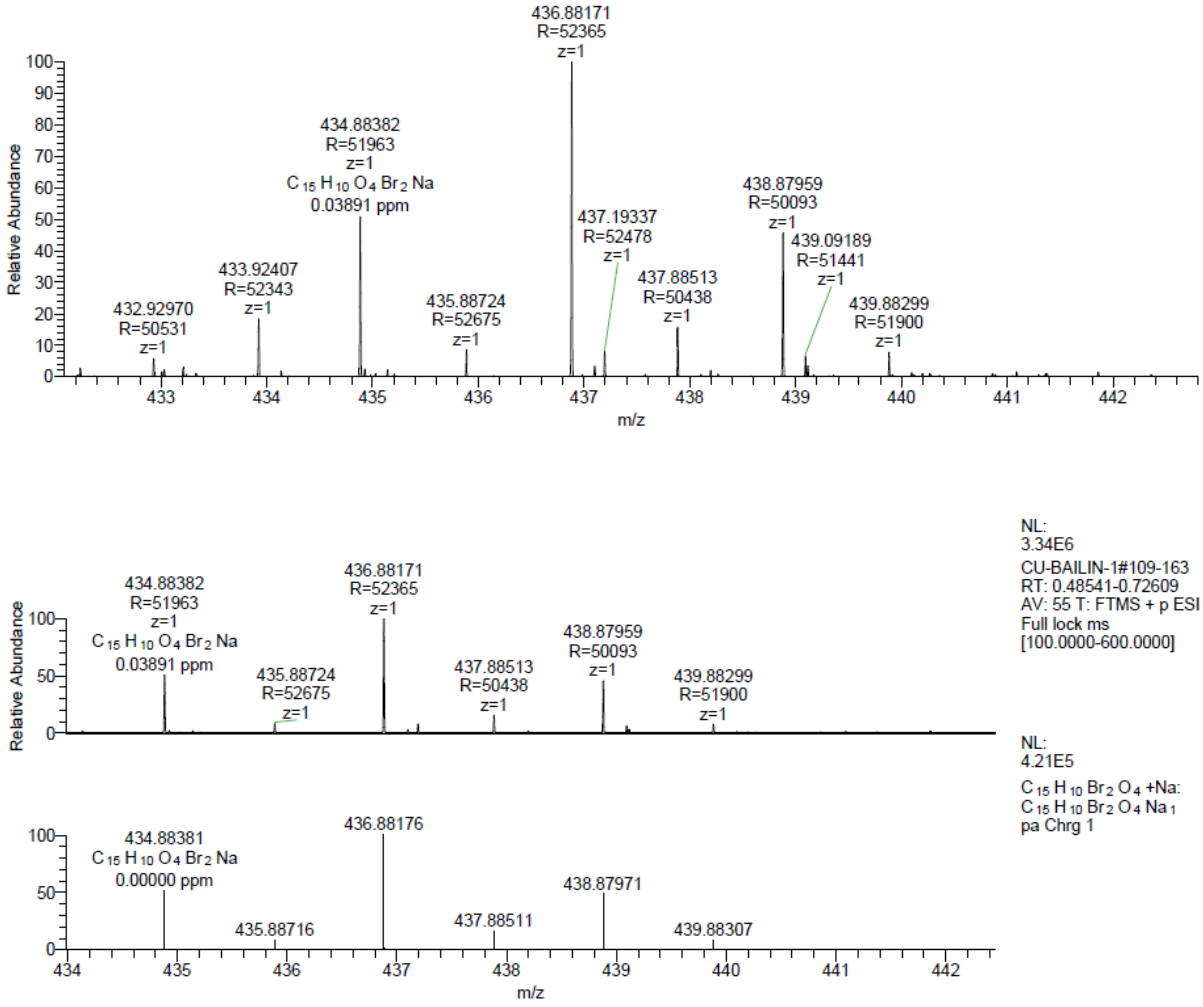
HRMS of **14**



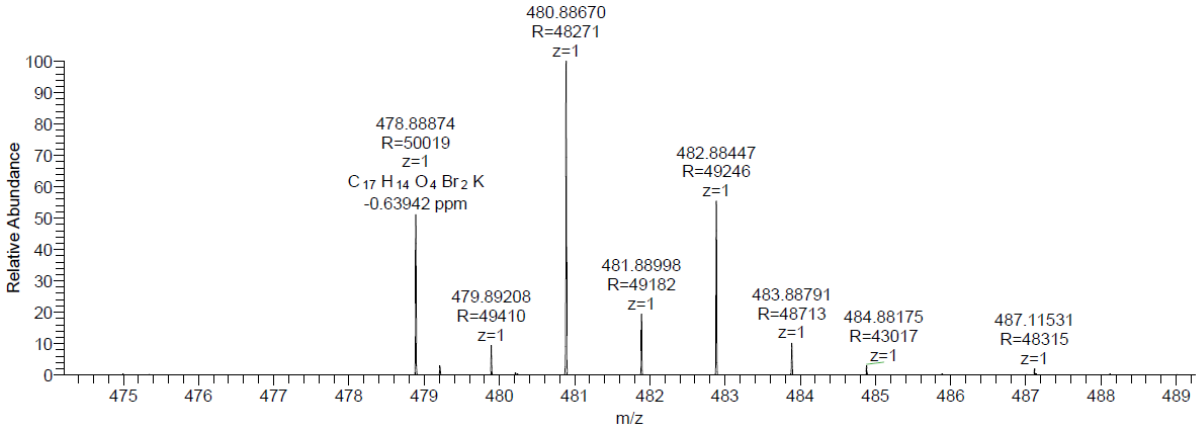
HRMS of **15**

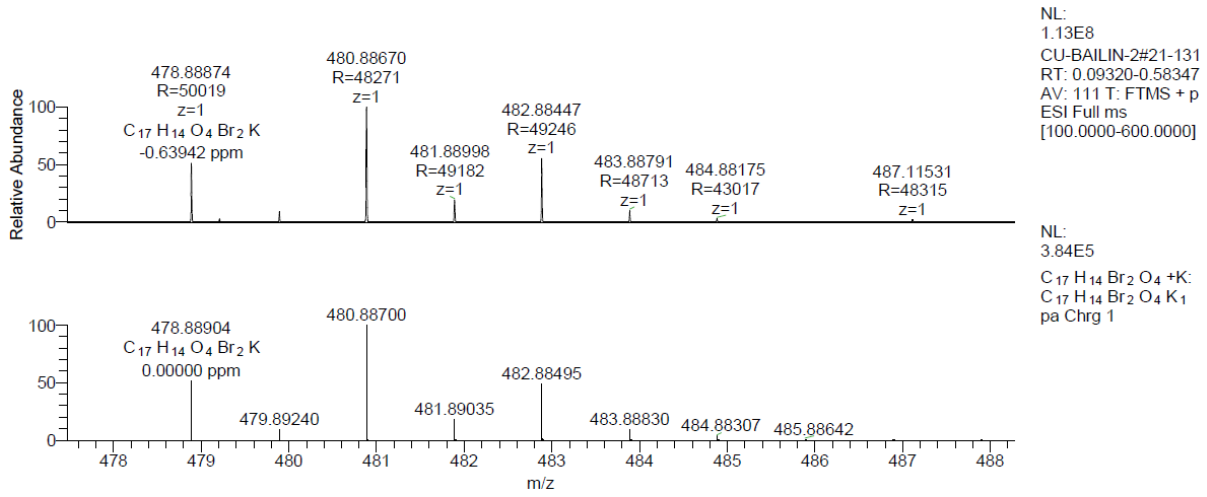


HRMS of **16**

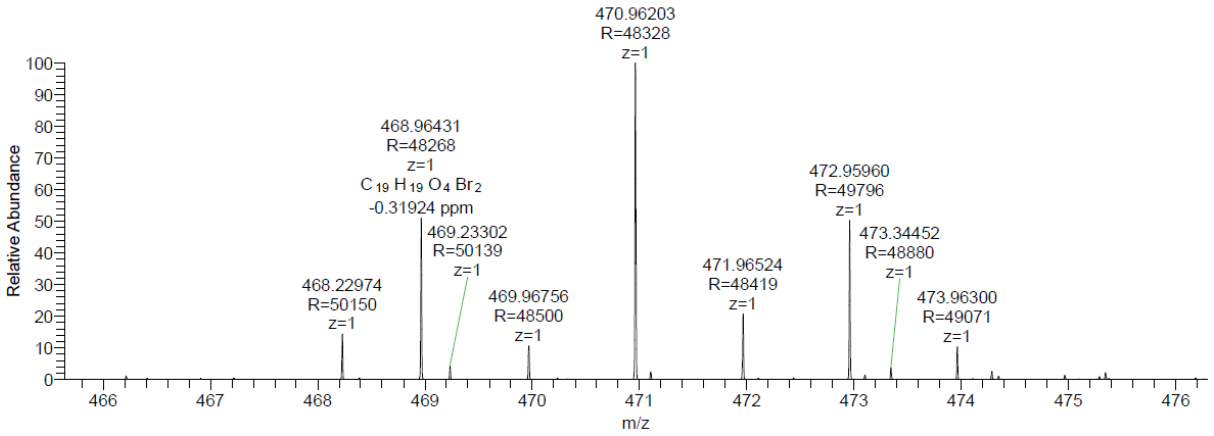


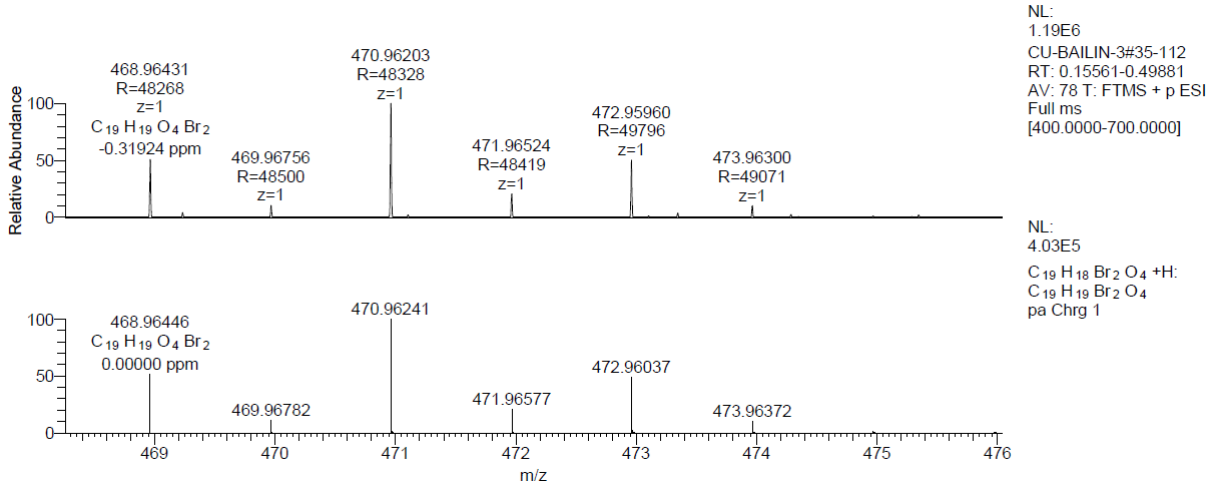
HRMS of **17**



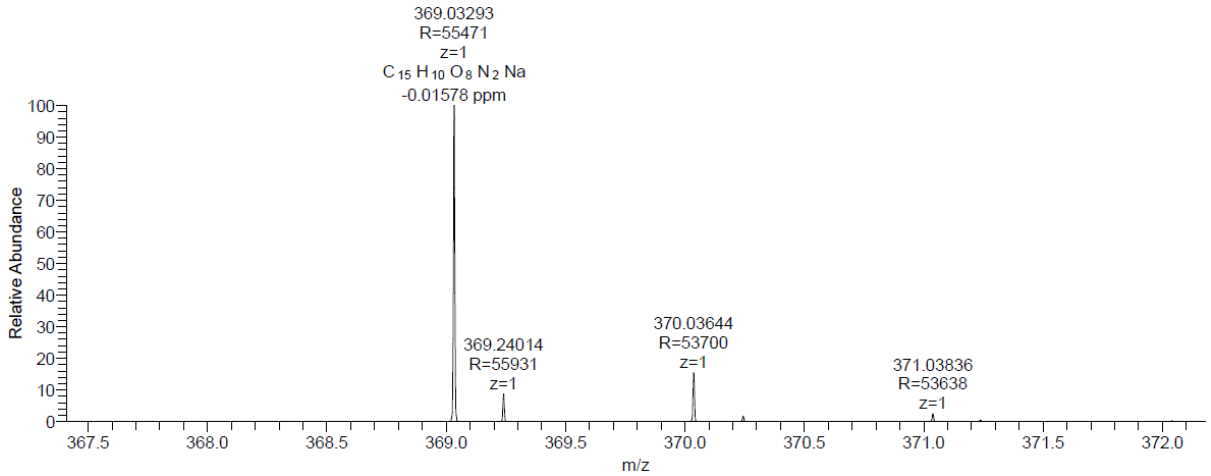

NL:
1.13E8
CU-BAILIN-2#21-131
RT: 0.09320-0.58347
AV: 111 T: FTMS + p
ESI Full ms
[100.0000-600.0000]
478.88874
R=50019
z=1
C17 H14 O4 Br2 K
-0.63942 ppm
480.88670
R=48271
z=1
481.88998
R=49182
z=1
482.88447
R=49246
z=1
483.88791
R=48713
z=1
484.88175
R=43017
z=1
487.11531
R=48315
z=1
Relative Abundance
NL:
3.84E5
C17 H14 Br2 O4 +K:
C17 H14 Br2 O4 K1
pa Chrg 1
478.88904
C17 H14 O4 Br2 K
0.00000 ppm
480.88700
479.89240
481.89035
482.88495
483.88830
484.88307
485.88642
m/z

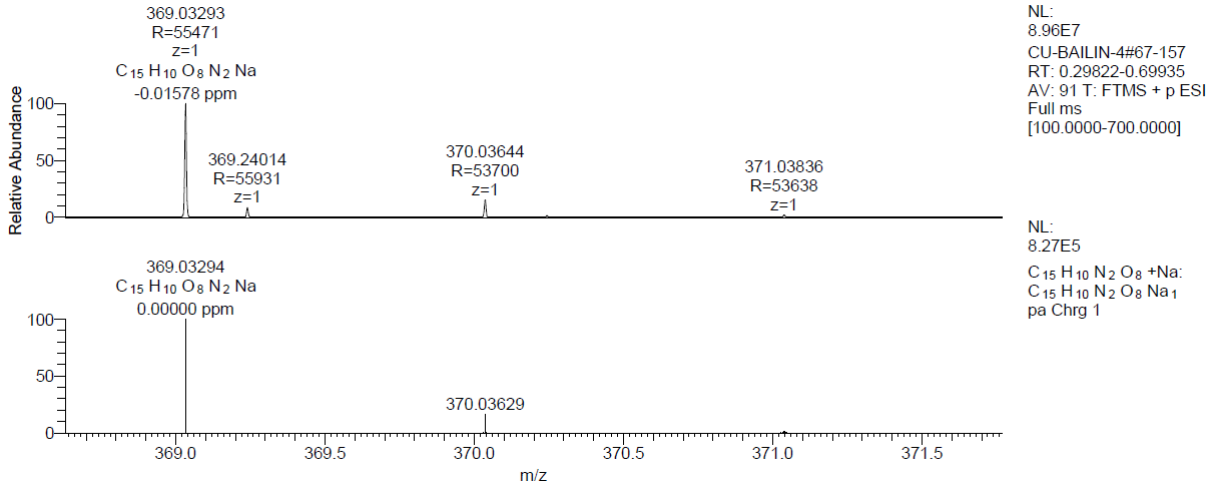
HRMS of **19**


470.96203
R=48328
z=1
468.96431
R=48268
z=1
C19 H19 O4 Br2
-0.31924 ppm
469.23302
R=50139
z=1
468.22974
R=50150
z=1
469.96756
R=48500
z=1
471.96524
R=48419
z=1
472.95960
R=49796
z=1
473.34452
R=48880
z=1
473.96300
R=49071
z=1
Relative Abundance
m/z

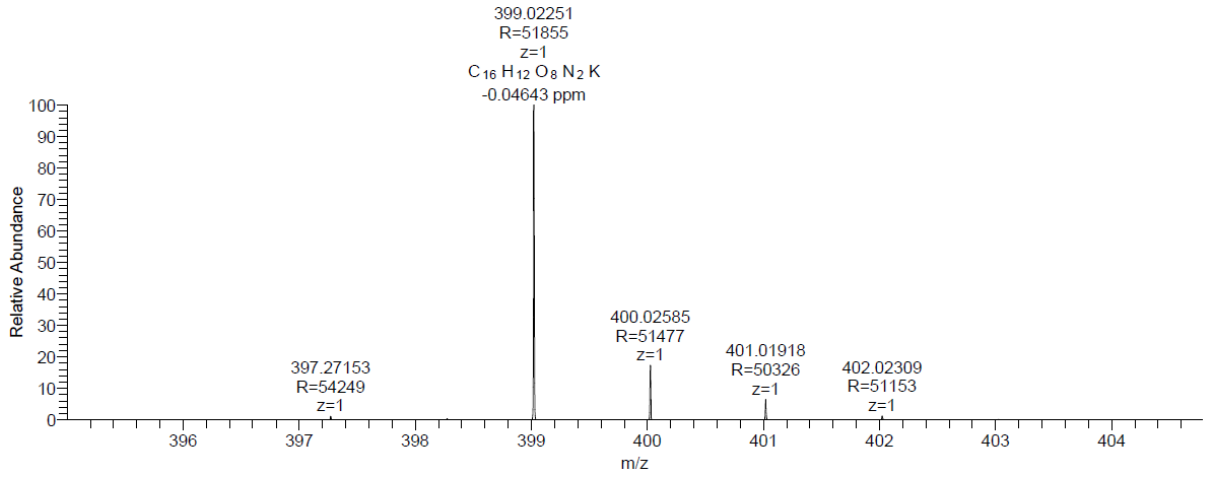

NL:
1.19E6
CU-BAILIN-3#35-112
RT: 0.15561-0.49881
AV: 78 T: FTMS + p ESI
Full ms
[400.0000-700.0000]
468.96431
R=48268
z=1
C19 H19 O4 Br2
-0.31924 ppm
469.96756
R=48500
z=1
470.96203
R=48328
z=1
471.96524
R=48419
z=1
472.95960
R=49796
z=1
473.96300
R=49071
z=1
Relative Abundance
NL:
4.03E5
C19 H18 Br2 O4 +H:
C19 H19 Br2 O4
pa Chrg 1
468.96446
C19 H19 O4 Br2
0.00000 ppm
469.96782
470.96241
471.96577
472.96037
473.96372
m/z

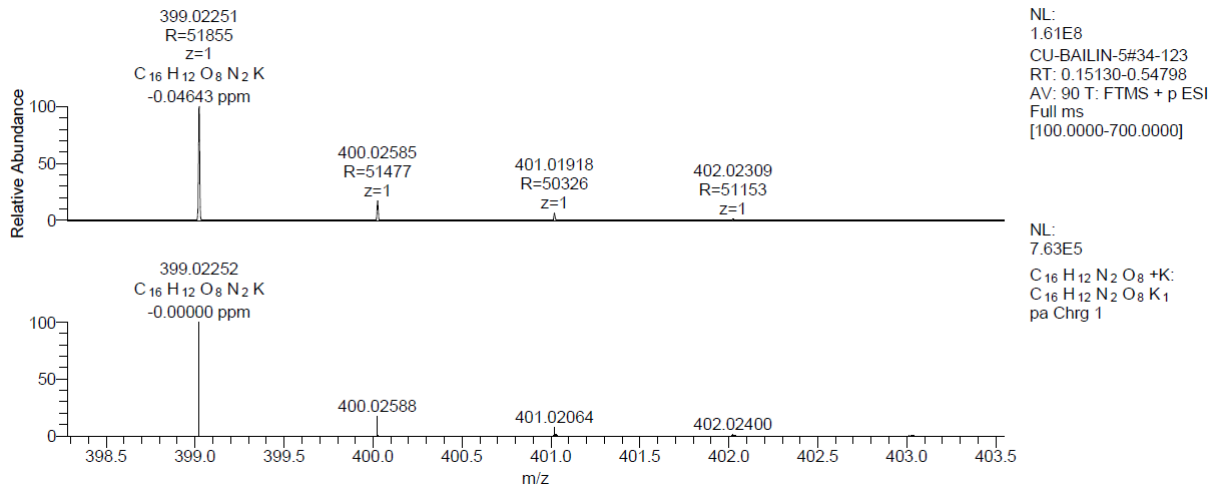
HRMS of **20**



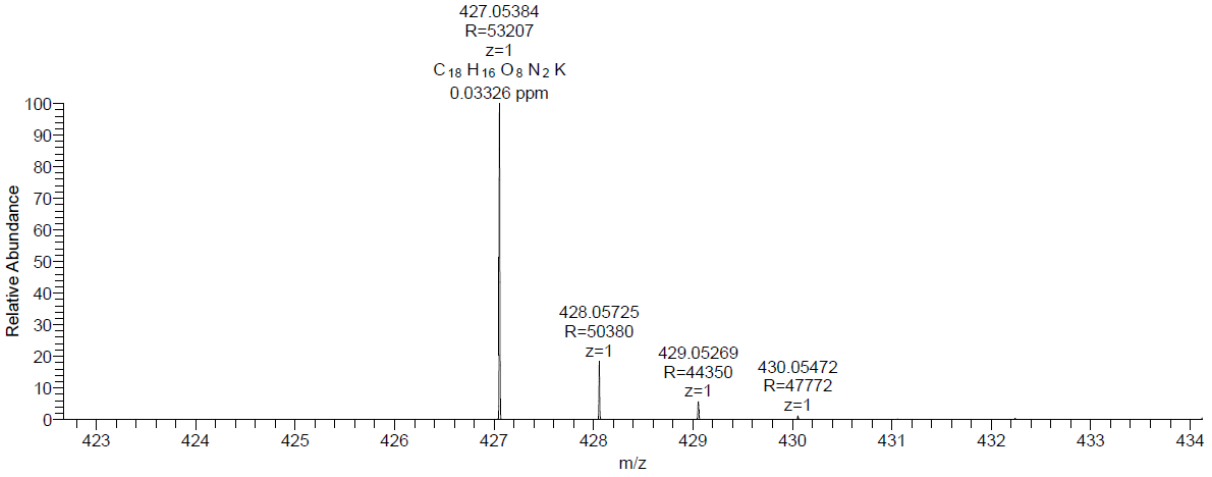


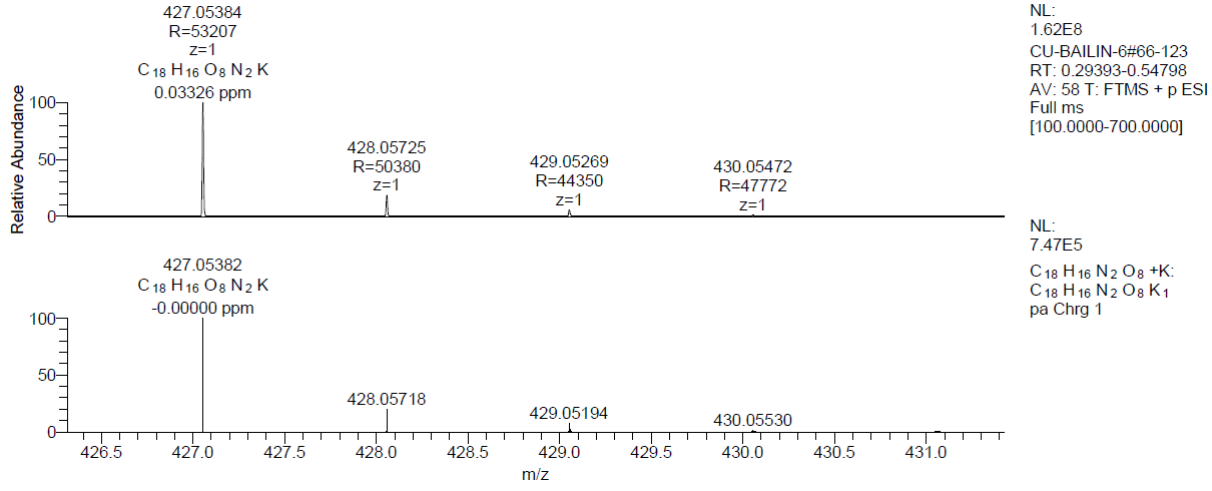
HRMS of **21**



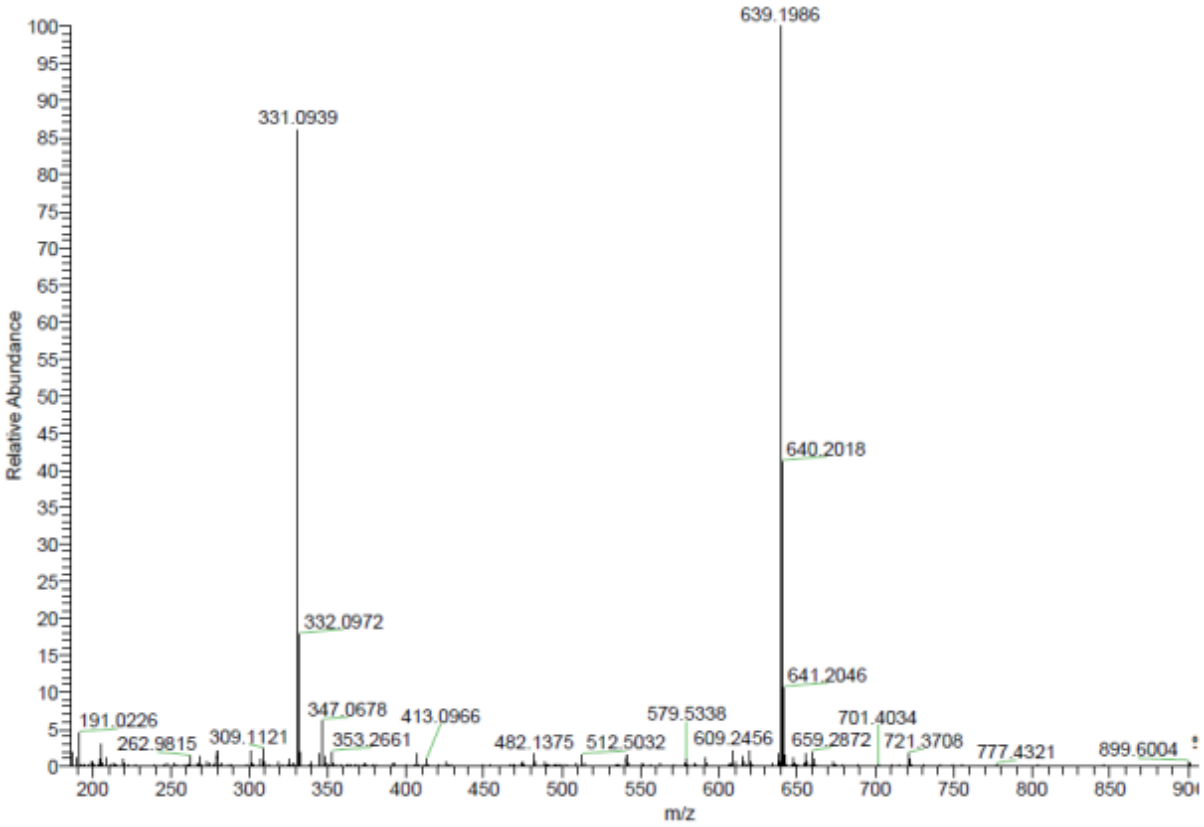

399.02251
R=51855
z=1
C 16 H 12 O 8 N 2 K
-0.04643 ppm
400.02585
R=51477
z=1
401.01918
R=50326
z=1
402.02309
R=51153
z=1
Relative Abundance
NL:
1.61E8
CU-BAILIN-5#34-123
RT: 0.15130-0.54798
AV: 90 T: FTMS + p ESI
Full ms
[100.0000-700.0000]
399.02252
C 16 H 12 O 8 N 2 K
-0.00000 ppm
400.02588
401.02064
402.02400
NL:
7.63E5
C 16 H 12 N 2 O 8 +K:
C 16 H 12 N 2 O 8 K 1
pa Chrg 1
m/z

HRMS of **22**

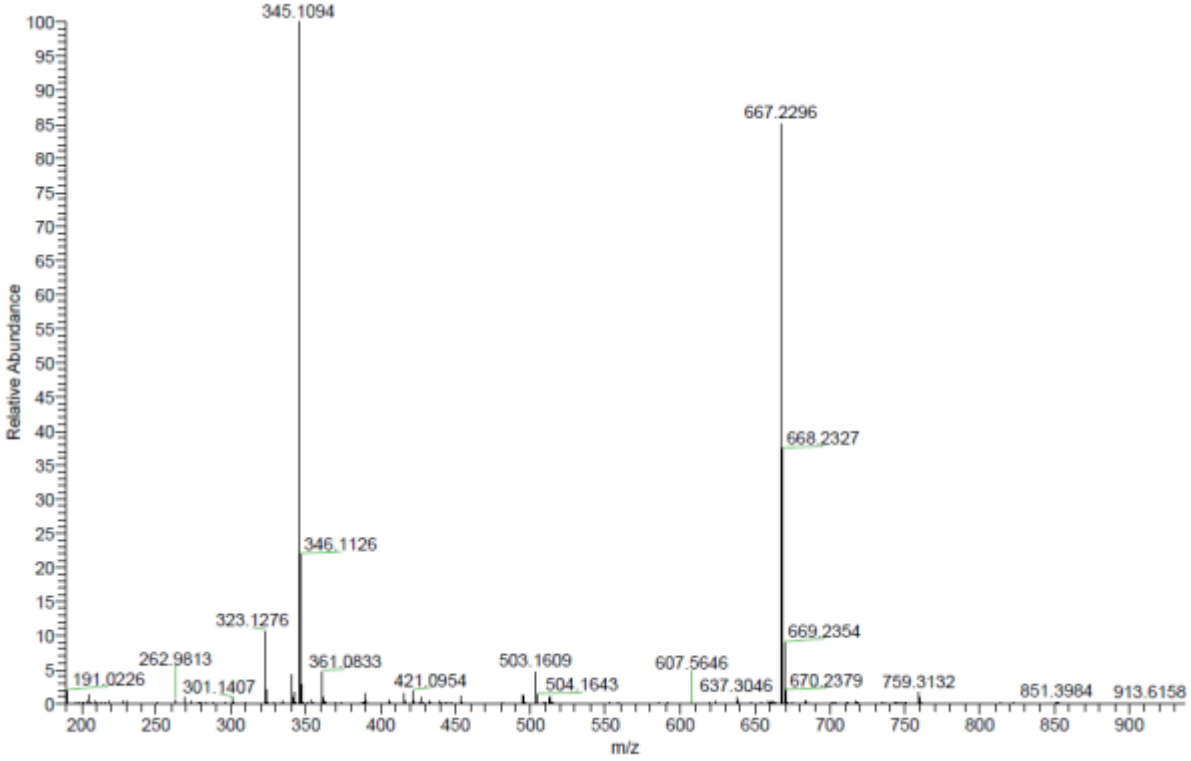

427.05384
R=53207
z=1
C 18 H 16 O 8 N 2 K
0.03326 ppm
428.05725
R=50380
z=1
429.05269
R=44350
z=1
430.05472
R=47772
z=1
Relative Abundance
m/z


427.05384
R=53207
z=1
C 18 H 16 O 8 N 2 K
0.03326 ppm
428.05725
R=50380
z=1
429.05269
R=44350
z=1
430.05472
R=47772
z=1
Relative Abundance
NL:
1.62E8
CU-BAILIN-6#66-123
RT: 0.29393-0.54798
AV: 58 T: FTMS + p ESI
Full ms
[100.0000-700.0000]
427.05382
C 18 H 16 O 8 N 2 K
-0.00000 ppm
428.05718
429.05194
430.05530
NL:
7.47E5
C 18 H 16 N 2 O 8 +K:
C 18 H 16 N 2 O 8 K 1
pa Chrg 1
m/z

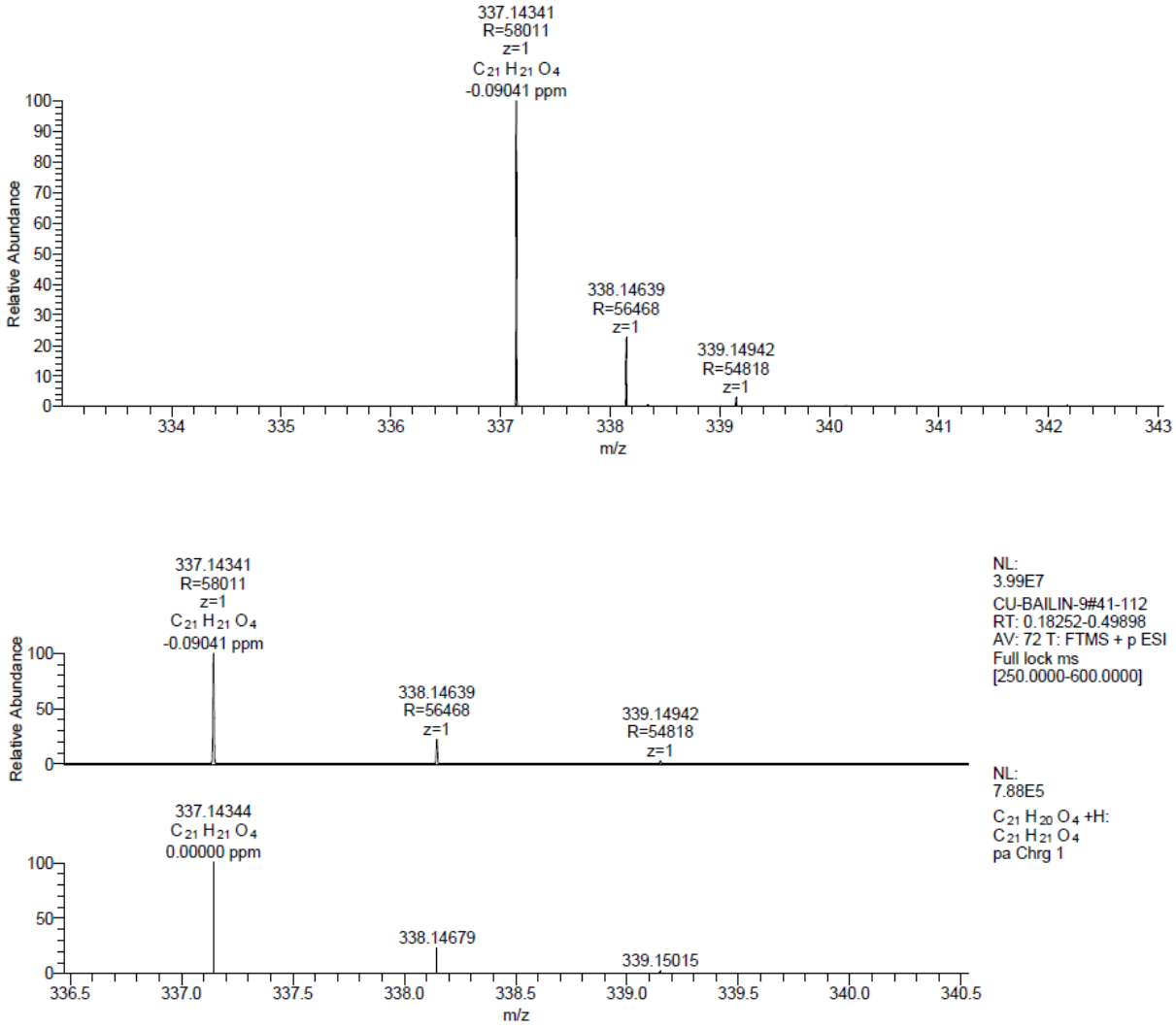
HRMS of **23**



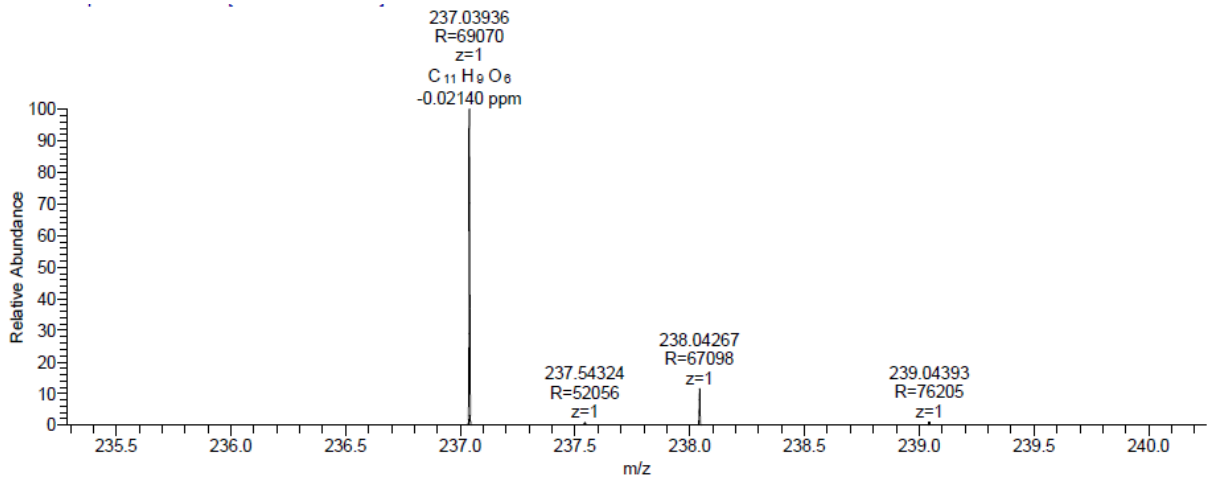
HRMS of **24**

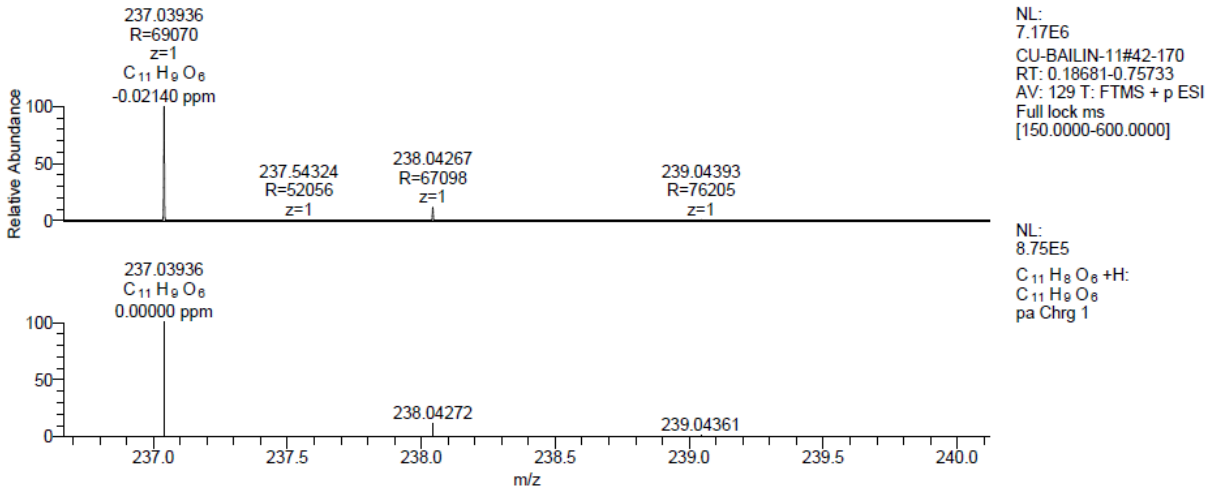


HRMS of **25**

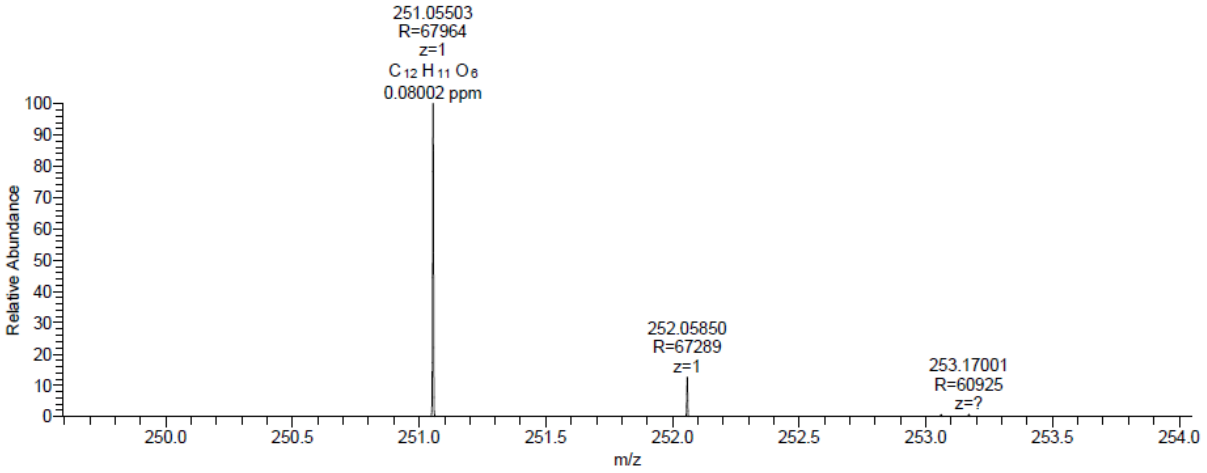


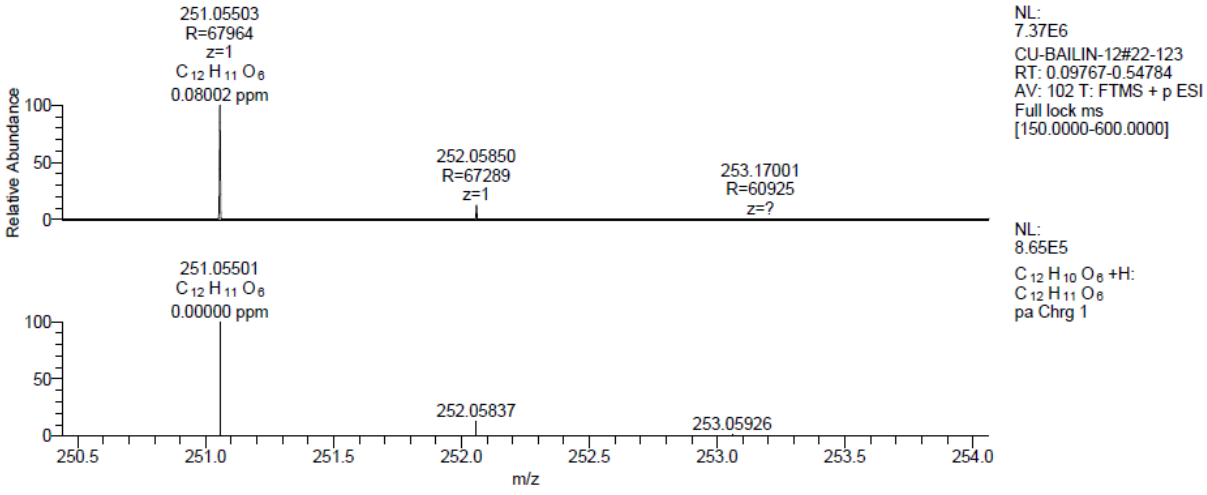
HRMS of **27**



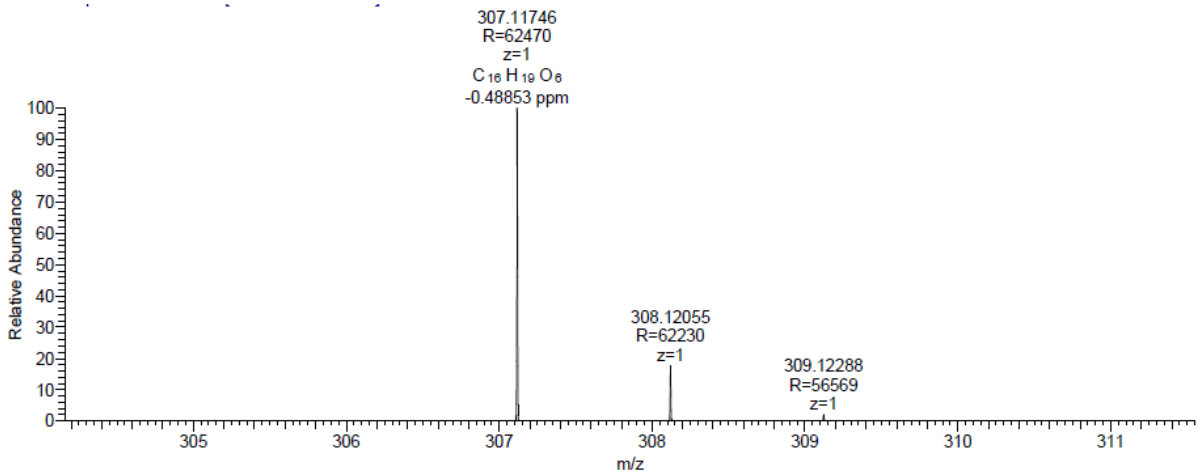

237.03936
R=69070
z=1
C 11 H 9 O 6
-0.02140 ppm
237.54324
R=52056
z=1
238.04267
R=67098
z=1
239.04393
R=76205
z=1
NL:
7.17E6
CU-BAILIN-11#42-170
RT: 0.18681-0.75733
AV: 129 T: FTMS + p ESI
Full lock ms
[150.0000-600.0000]
237.03936
C 11 H 9 O 6
0.00000 ppm
238.04272
239.04361
NL:
8.75E5
C 11 H 8 O 6 +H:
C 11 H 9 O 6
pa Chrg 1
Relative Abundance
m/z

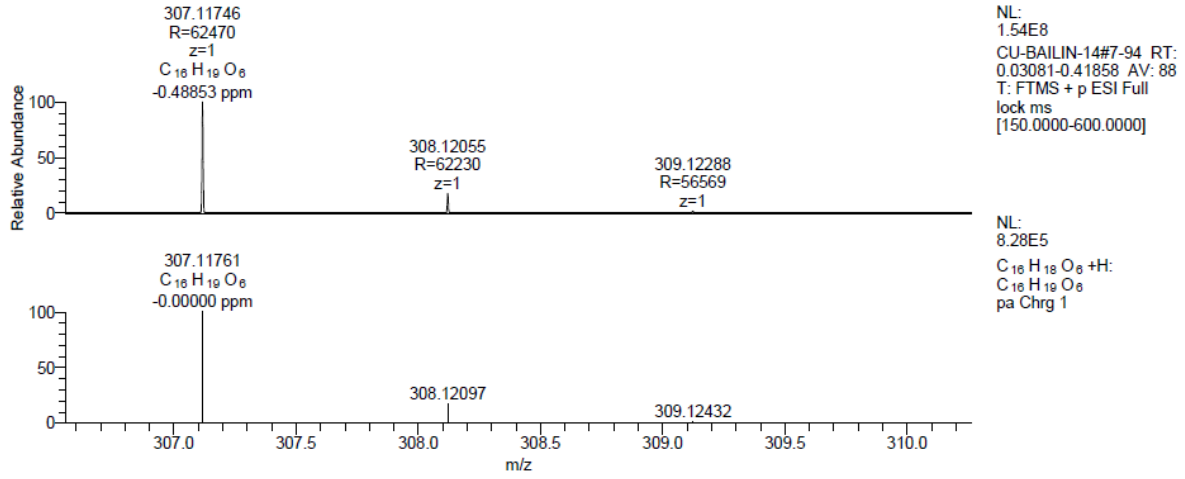
HRMS of **28**


251.05503
R=67964
z=1
C 12 H 11 O 6
0.08002 ppm
252.05850
R=67289
z=1
253.17001
R=60925
z=?
Relative Abundance
m/z

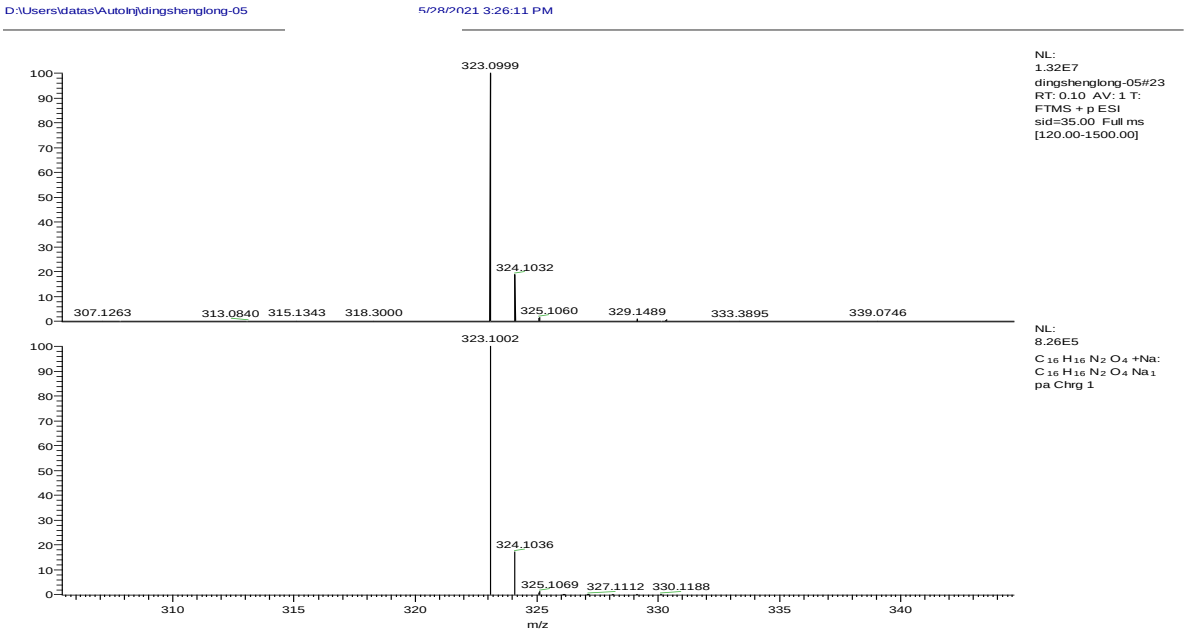

251.05503
R=67964
z=1
C 12 H 11 O 6
0.08002 ppm
252.05850
R=67289
z=1
253.17001
R=60925
z=?
NL:
7.37E6
CU-BAILIN-12#22-123
RT: 0.09767-0.54784
AV: 102 T: FTMS + p ESI
Full lock ms
[150.0000-600.0000]
251.05501
C 12 H 11 O 6
0.00000 ppm
252.05837
253.05926
NL:
8.65E5
C 12 H 10 O 6 +H:
C 12 H 11 O 6
pa Chrg 1
Relative Abundance
m/z

HRMS of **30**

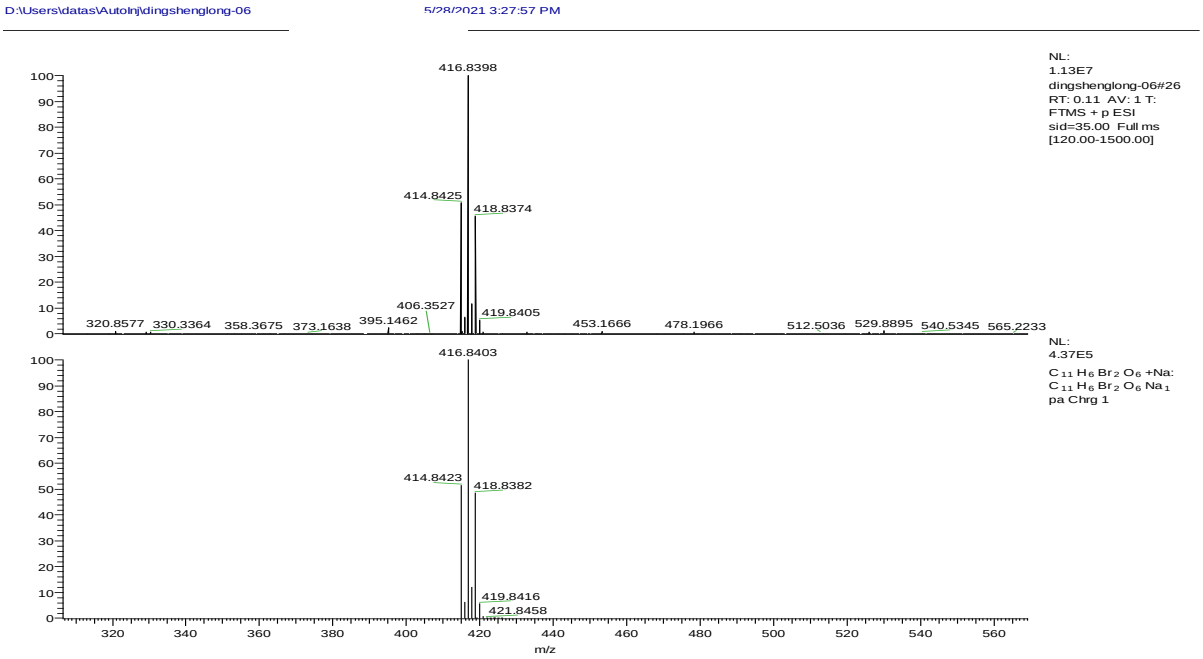




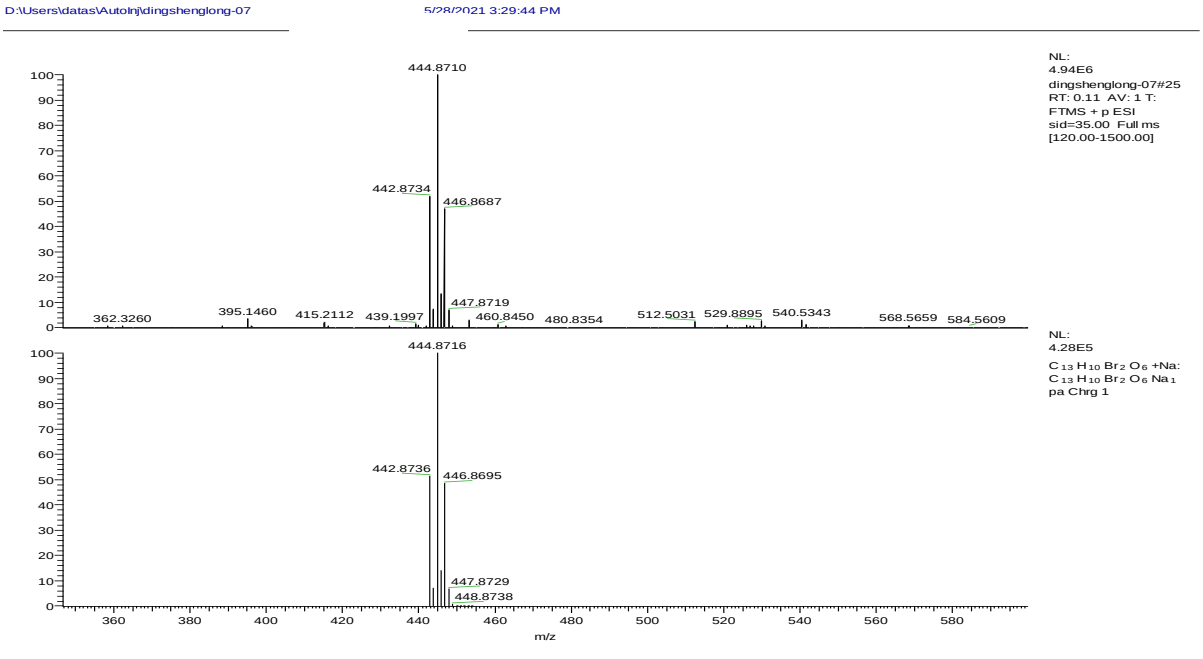
HRMS of **31**



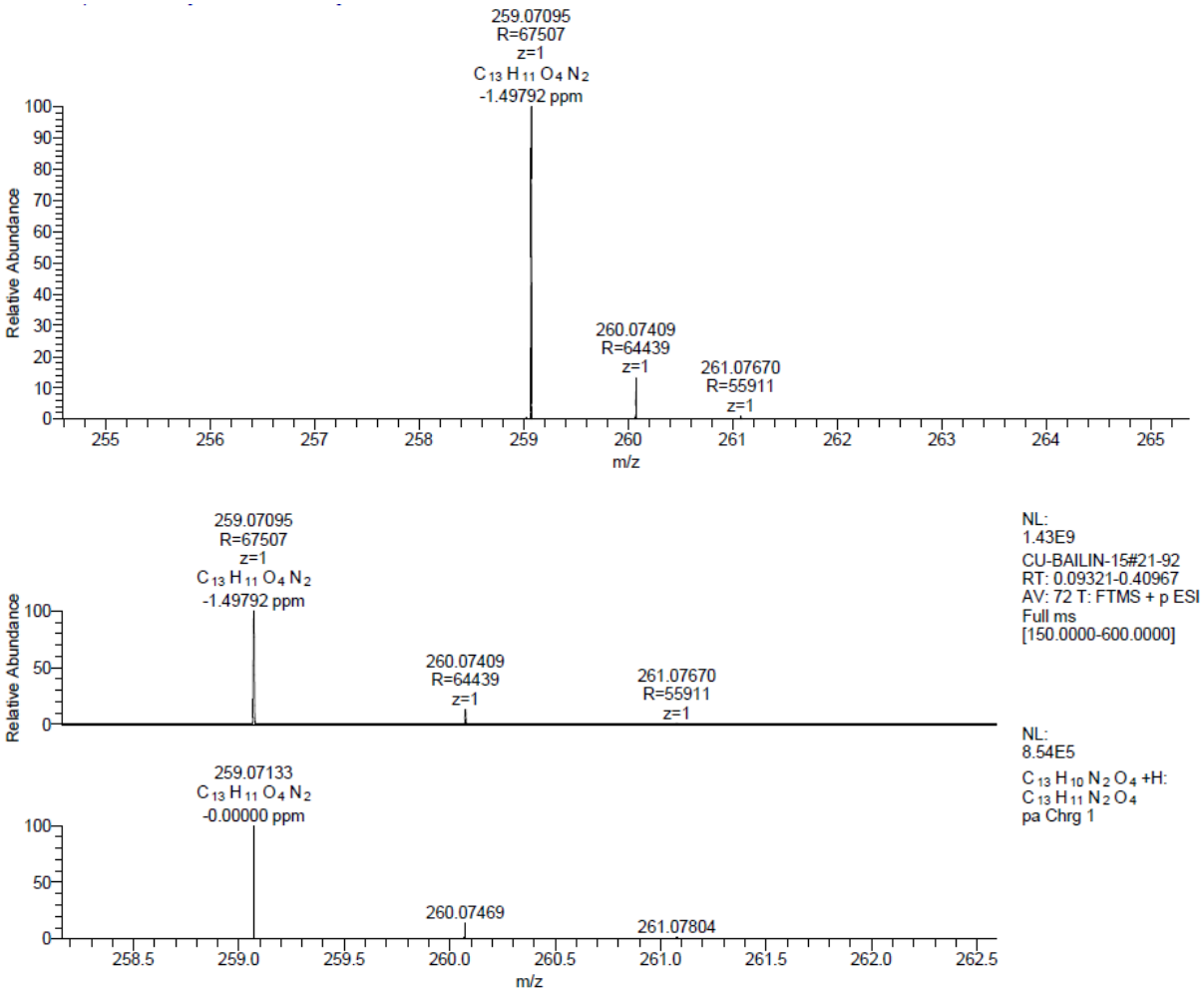
HRMS of **32**



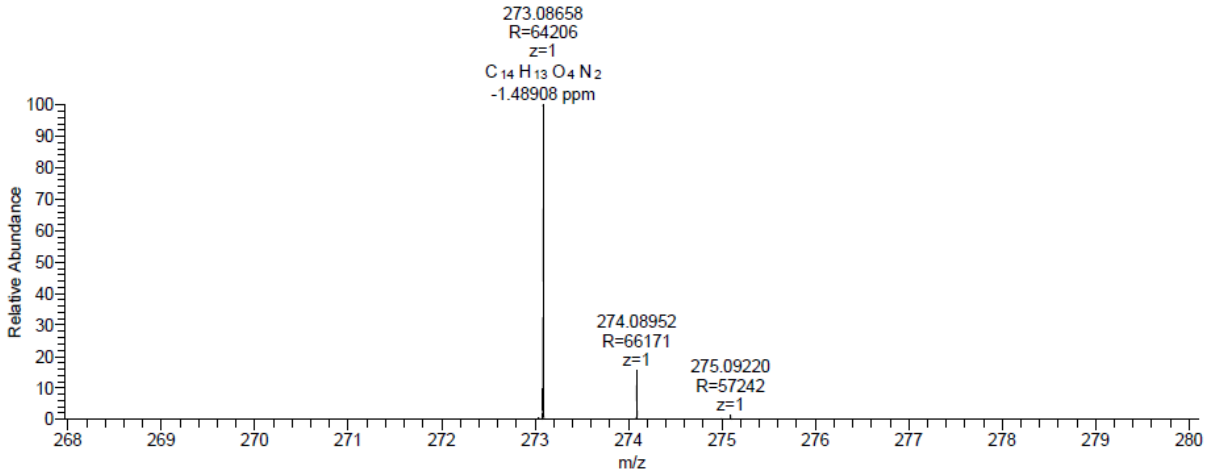
HRMS of **33**

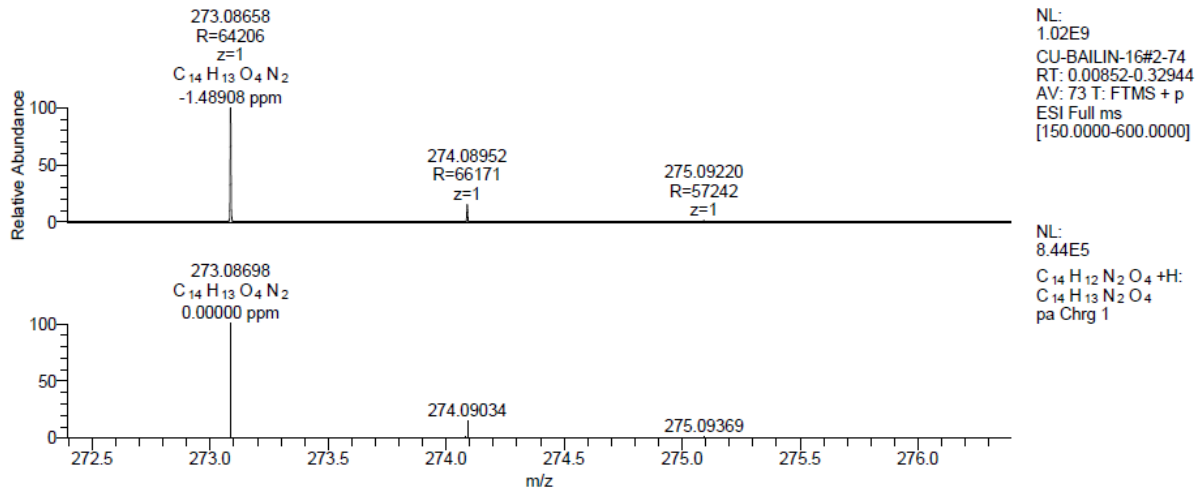


HRMS of **34**

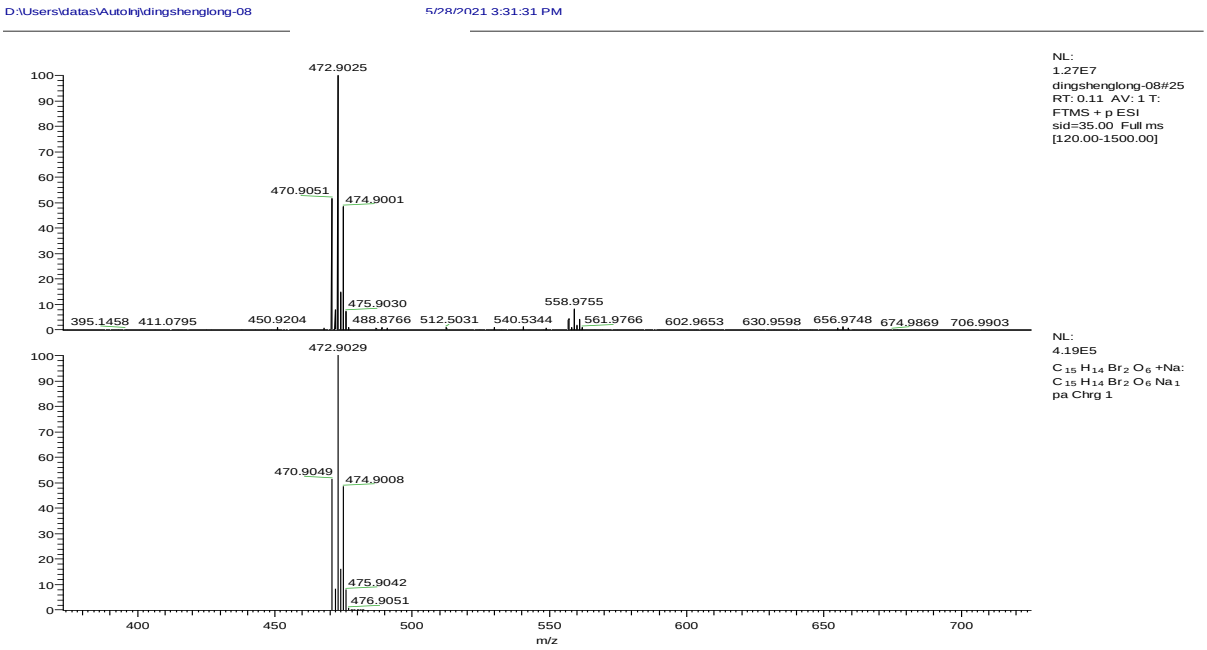


HRMS of **35**




273.08658
R=64206
z=1
C 14 H 13 O 4 N 2
-1.48908 ppm
274.08952
R=66171
z=1
275.09220
R=57242
z=1
Relative Abundance
NL:
1.02E9
CU-BAILIN-16#2-74
RT: 0.00852-0.32944
AV: 73 T: FTMS + p
ESI Full ms
[150.0000-600.0000]
273.08698
C 14 H 13 O 4 N 2
0.00000 ppm
274.09034
275.09369
NL:
8.44E5
C 14 H 12 N 2 O 4 +H:
C 14 H 13 N 2 O 4
pa Chrg 1
m/z

HRMS of **36**


D:\Users\datas\AutoIrj\dingshenglong-08
5/28/2021 3:31:31 PM
472.9025
470.9051
474.9001
475.9030
558.9755
395.1458
411.0795
450.9204
488.8766
512.5031
540.5344
561.9766
602.9653
630.9598
656.9748
674.9869
706.9903
NL:
1.27E7
dingshenglong-08#25
RT: 0.11 AV: 1 T:
FTMS + p ESI
sid=35.00 Full ms
[120.00-1500.00]
472.9029
470.9049
474.9008
475.9042
476.9051
NL:
4.19E5
C 15 H 14 Br 2 O 6 +Na:
C 15 H 14 Br 2 O 6 Na 1
pa Chrg 1
m/z