Supporting Information

Copper/Iron Controlled Regioselective 1,2-Carboazidation of 1,3-Dienes with Acetonitrile and Azidotrimethylsilane

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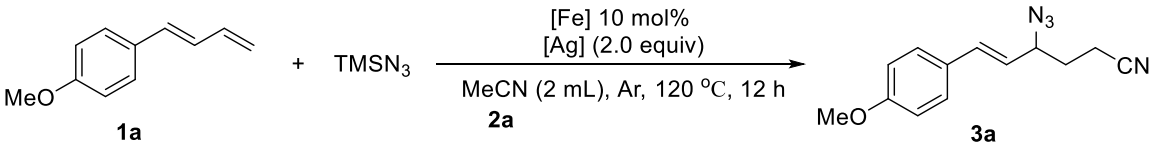
(A) Typical experimental procedure

(a) General Information

The ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ solvents on a NMR spectrometer using TMS as the internal standard. HRMS was measured on an electrospray ionization (ESI) apparatus using time-of-flight (TOF) mass spectrometry.

General procedure for synthesis of compound 3a.

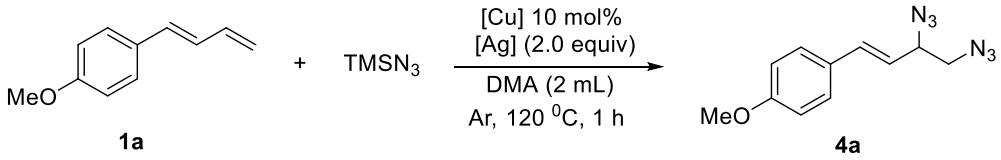


To a Schlenk tube were added substrates **1a** (0.2 mmol), $TMSN_3$ (0.4 mmol, 2.0 equiv), $FeCl_2$ (0.02 mmol, 10 mol%), Ag_2CO_3 (0.4 mmol, 2.0 equiv) and MeCN (2 mL), the tube was then charged with Ar. The mixture was stirred at 120 °C until complete consumption of starting material as monitored by TLC (about 12 h). After the reaction was finished, the reaction mixture was concentrated in vacuum, and the resulting residue was purified by silica gel column chromatography (petroleum ether/ethyl acetate) to afford the desired product.

One millimol reaction： To a Schlenk tube were added substrates **1a** (1 mmol), $TMSN_3$ (2 mmol, 2.0 equiv), $FeCl_2$ (0.1 mmol, 10 mol%), Ag_2CO_3 (2 mmol, 2.0 equiv) and MeCN (10 mL), the tube was then charged with Ar. The mixture was stirred at 120 °C until complete consumption of starting material as monitored by TLC (about 12 h). After the reaction was finished, the reaction mixture was concentrated in vacuum,

and the resulting residue was purified by silica gel column chromatography (petroleum ether/ethyl acetate) to afford the desired product **3a** (76%, 183.9 mg).

General procedure for synthesis of compound 4a.

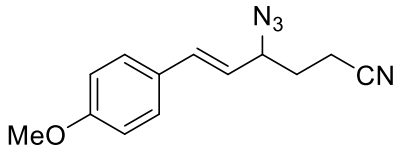


To a Schlenk tube were added substrates **1a** (0.2 mmol), $TMSN_3$ (0.6 mmol, 3.0 equiv), CuSCN (0.02 mmol, 10 mol%), Ag_2CO_3 (0.4 mmol, 2.0 equiv) and DMA (2 mL), the tube was then charged with Ar. The mixture was stirred at 120 °C until complete consumption of starting material as monitored by TLC (about 1 h). After the reaction was finished, the reaction mixture was concentrated in vacuum, and the resulting residue was purified by silica gel column chromatography (petroleum ether/ethyl acetate) to afford the desired product.

One millimol reaction： To a Schlenk tube were added substrates **1a** (1 mmol), $TMSN_3$ (3 mmol, 3.0 equiv), CuSCN (0.1 mmol, 10 mol%), Ag_2CO_3 (2 mmol, 2.0 equiv) and DMA (10 mL), the tube was then charged with Ar. The mixture was stirred at 120 °C until complete consumption of starting material as monitored by TLC (about 1 h). After the reaction was finished, the reaction mixture was concentrated in vacuum, and the resulting residue was purified by silica gel column chromatography (petroleum ether/ethyl acetate) to afford the desired product **4a** (95%, 231.8 mg).

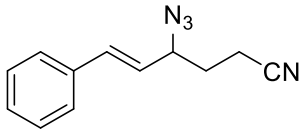
（B）Analytical data

(*E*)-4-azido-6-(4-methoxyphenyl)hex-5-enenitrile (3a):



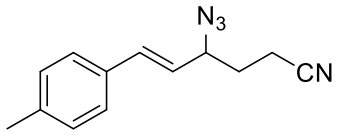
36.8 mg, 76% yield; Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.34 (d, *J* = 8.5 Hz, 2H), 6.87 (d, *J* = 8.5 Hz, 2H), 6.63 (d, *J* = 16.0 Hz, 1H), 5.95 – 5.89 (m, 1H), 4.15 (q, *J* = 7.5 Hz, 1H), 3.79 (s, 3H), 2.45 - 2.41 (m, 2H), 1.90 - 1.86 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.8 , 134.5 , 127.9 , 127.8 , 122.2 , 118.8 , 114.0 , 63.1 , 55.1 , 30.3 , 13.8. HRMS *m/z* (ESI) calcd for $C_{13}H_{15}N_4O([M+H]^+)$ 243.1240, found 243.1240.

(*E*)-1-(hept-1-en-6-yn-1-yl)-4-methoxybenzene (3b):



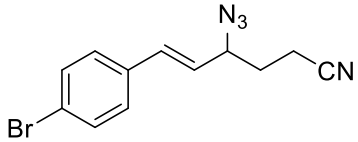
29.3 mg, 69% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.42 (d, *J* = 7.0 Hz, 2H), 7.35 (t, *J* = 7.5 Hz, 2H), 7.30 (t, *J* = 7.5 Hz, 1H), 6.71 (d, *J* = 16.0 Hz, 1H), 6.11 – 6.05 (m, 1H), 4.24 – 4.19 (m, 1H), 2.52 – 2.43 (m, 2H), 1.95 – 1.90 (m, 2H).^{13}C NMR (125 MHz, Chloroform-*d*) δ 135.3 , 135.1 , 128.7 , 128.6 , 126.8 , 124.7 , 118.7 , 63.0 , 30.4 , 14.0. HRMS *m/z* (ESI) calcd. for $C_{12}H_{13}N_4([M+H]^+)$213.1135, found 213.1135.

(*E*)-4-azido-6-(p-tolyl)hex-5-enenitrile (3c):



28.9 mg, 64% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.31 (d, *J* = 8.0 Hz, 2H), 7.16 (d, *J* = 7.5 Hz, 2H), 6.68 (d, *J* = 15.5 Hz, 1H), 6.05 – 5.99 (m, 1H), 4.23 – 4.17 (m, 1H), 2.50 – 2.45 (m, 2H), 2.36 (s, 3H), 1.95 – 1.89 (m, 2H).^{13}C NMR (125 MHz, Chloroform-*d*) δ 138.7 , 135.1 , 132.5 , 129.4 , 126.7 , 123.6 , 118.8 , 63.1 , 30.5 , 21.3 , 14.0. HRMS *m/z* (ESI) calcd for $C_{13}H_{15}N_4([M+H]^+)$ 227.1291, found 227.1291.

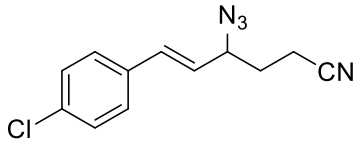
(*E*)-4-azido-6-(4-bromophenyl)hex-5-enenitrile (3d):



31.3 mg, 54% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.41 (d, *J* = 8.0 Hz, 2H), 7.22 – 7.19 (m, 2H), 6.58 (d, *J* = 16.0 Hz, 1H), 6.03 – 5.97 (m, 1H), 4.17 – 4.11 (m, 1H), 2.46 – 2.37 (m, 2H), 1.88 – 1.83 (m, 2H).^{13}C NMR (125 MHz,

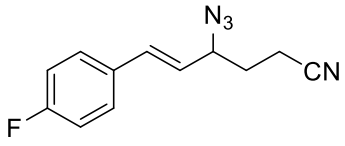
Chloroform-*d*) δ 134.2 , 133.8 , 131.9 , 128.3 , 125.5 , 122.5 , 118.7 , 62.9 , 30.4 , 13.9. HRMS *m/z* (ESI) calcd. for $C_{12}H_{12}BrN_4$([M+H]$^+$)291.0240, found 291.0240.

(*E*)-4-azido-6-(4-chlorophenyl)hex-5-enenitrile (3e):



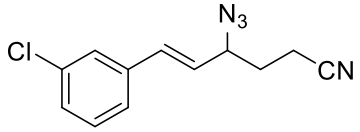
36.4 mg, 74% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.36 – 7.30 (m, 4H), 6.67 (d, *J* = 15.5 Hz, 1H), 6.09 – 6.03 (m, 1H), 4.24 – 4.19 (m, 1H), 2.53 – 2.44 (m, 2H), 1.95 – 1.90 (m, 2H).^{13}C NMR (125 MHz, Chloroform-*d*) δ 134.3 , 133.8 , 133.7 , 128.9 , 128.0 , 125.4 , 118.7 , 62.9 , 30.4 , 13.9. HRMS *m/z* (ESI) calcd.for $C_{12}H_{12}ClN_4$ ([M+H]$^+$) 247.0745, found 247.0745.

(*E*)-4-azido-6-(4-fluorophenyl)hex-5-enenitrile (3f):



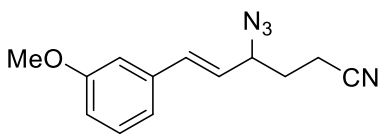
33.6 mg, 73% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.32 (t, *J* = 7.0 Hz, 2H), 6.97 (t, *J* = 8.5 Hz, 2H), 6.61 (d, *J* = 16.0 Hz, 1H), 5.96 – 5.90 (m, 1H), 4.17 – 4.11 (m, 1H), 2.46 – 2.37 (m, 2H), 1.88 – 1.83 (m, 2H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 163.9, 161.9, 134.0, 131.5, 131.4, 128.5, 128.4, 124.5, 124.5, 118.7, 115.8, 115.7, 63.0, 30.4, 14.0. ^{19}F NMR (471 MHz, Chloroform-d) δ -112.59. HRMS *m/z* (ESI) calcd. for $C_{12}H_{12}FN_4$([M+H]$^+$) 231.1041, found 231.1042.

(*E*)-4-azido-6-(3-chlorophenyl)hex-5-enenitrile (3g):



36.4 mg, 74% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.40 (s, 1H), 7.27 (s, 3H), 6.65 (d, *J* = 16.0 Hz, 1H), 6.12 – 6.06 (m, *J* = 16.0, 8.0 Hz, 1H), 4.24 – 4.18 (m, 1H), 2.50 – 2.45 (m, 2H), 1.94 – 1.89 (m, 2H).^{13}C NMR (125 MHz, Chloroform-*d*) δ 137.1 , 134.6 , 133.5 , 129.9 , 128.4 , 126.5 , 126.3 , 125.0 , 118.6 , 62.7 , 30.2 , 13.8. HRMS *m/z* (ESI) calcd. for $C_{12}H_{12}ClN_4$([M+H]$^+$) 247.0745, found 247.0745.

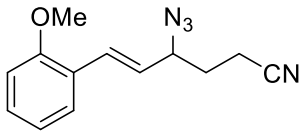
(*E*)-4-azido-6-(3-methoxyphenyl)hex-5-enenitrile (3h):



27.1 mg, 56% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.26 (t, *J* = 5.0 Hz, 1H), 7.01 (d, *J* = 7.0 Hz, 1H), 6.94 (s, 1H), 6.86 (d, *J* = 8.0 Hz, 1H), 6.68 (d, *J* = 15.5 Hz, 1H), 6.10 – 6.04 (m, 1H), 4.24 – 4.18 (m, 1H), 3.83 (s, 3H),

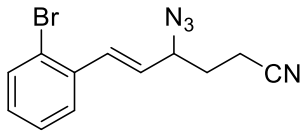
2.53 – 2.43 (m, 2H), 1.95 – 1.90 (m, 2H). ^{13}C NMR (125 MHz, Chloroform-d) δ 159.9, 136.7, 135.0, 129.7, 125.0, 119.4, 118.7, 114.2, 112.1, 63.0, 55.3, 30.4, 14.0. HRMS *m/z* (ESI) calcd. for $C_{13}H_{15}N_4O([M+H]^+)$ 243.1240, found 243.1236.

(*E*)-4-azido-6-(2-methoxyphenyl)hex-5-enenitrile (3i):



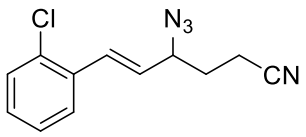
40.2 mg, 83% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.44 (d, *J* = 7.5 Hz, 1H), 7.30 – 7.26 (m, 1H), 7.01 (d, *J* = 16.0 Hz, 1H), 6.94 (t, *J* = 7.5 Hz, 1H), 6.89 (d, *J* = 8.5 Hz, 1H), 6. 14 – 6.08 (m, 1H), 4.20 – 4.17 (m, 1H), 3.86 (s, 3H), 2.50 – 2.44 (m, 2H), 1.95 – 1.90 (m, 2H).^{13}C NMR (125 MHz, Chloroform-*d*) δ 157.0 , 130.3 , 129.7 , 127.3 , 125.1 , 124.2 , 120.6 , 118.8 , 111.0 , 63.5 , 55.4 , 30.5 , 13.9. HRMS *m/z* (ESI) calcd. for $C_{13}H_{15}N_4O$ ($[M+H]^+$) 243.1240, found 243.1240.

(*E*)-4-azido-6-(2-bromophenyl)hex-5-enenitrile (3j):



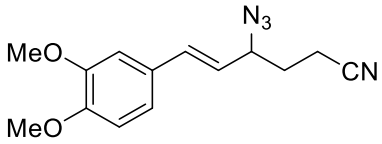
45.2 mg, 78% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.51 (d, *J* = 8.0 Hz, 1H), 7.45 (d, *J* = 8.0 Hz, 1H), 7.23 (t, *J* = 7.5 Hz, 1H), 7.09 (t, *J* = 8.0 Hz, 1H), 6.98 (d, *J* = 16.0 Hz, 1H), 5.98 – 5.92 (m, 1H), 4.23 – 4.17 (m, 1H), 2.43 (t, *J* = 7.5 Hz, 2H), 1.93 – 1.84 (m, 2H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 135.5, 133.9, 133.1, 129.8, 127.7, 127.6, 127.3, 123.8, 118.7, 62.7, 30.3, 13.9. HRMS *m/z* (ESI) calcd. for $C_{12}H_{12}BrN_4([M+H]^+)$ 291.0240, 291.0252.

(*E*)-4-azido-6-(2-chlorophenyl)hex-5-enenitrile (3k):



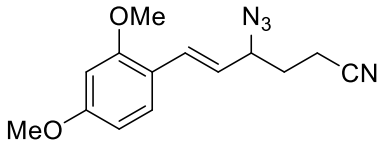
28.1 mg, 57% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.56 – 7.51 (m, 1H), 7.38 (d, *J* = 9.0 Hz, 1H), 7.26 – 7.23 (m, 2H), 7.09 (d, *J* = 16.0 Hz, 1H), 6.09 – 6.03 (m, 1H), 4.28 – 4.23 (m, 1H), 2.49 (t, *J* = 6.5 Hz, 2H), 1.97 – 1.92 (m, 2H).^{13}C NMR (125 MHz, Chloroform-*d*) δ 133.5 , 133.3 , 131.2 , 129.8 , 129.5 , 127.5 , 127.0 , 126.9 , 118.7 , 62.7 , 30.3 , 13.8. HRMS *m/z* (ESI) calcd for $C_{12}H_{12}ClN_4$ ($[M+H]^+$) 247.0745, found 247.0744.

(*E*)-4-azido-6-(3,4-dimethoxyphenyl)hex-5-enenitrile (3l):



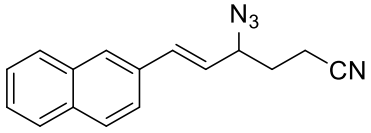
35.5 mg, 65% yield; Light red liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 6.97 (d, *J* = 9.0 Hz, 2H), 6.84 (d, *J* = 8.0 Hz, 1H), 6.64 (d, *J* = 15.5 Hz, 1H), 5.97 – 5.92 (m, 1H), 4.22 – 4.17 (m, 1H), 3.91 (s, 3H), 3.89 (s, 3H), 2.49 – 2.45 (m, 2H), 1.95 – 1.89 (m, 2H). ^{13}C NMR (125 MHz, Chloroform-d) δ 149.5, 149.0, 134.8, 128.2, 122.5, 120.2, 118.8, 111.0, 108.9, 63.1, 55.8, 55.8, 30.4, 13.9. HRMS *m/z* (ESI) calcd. for $C_{14}H_{17}N_4O_2$([M+H]$^+$) 273.1346, found 273.1346.

(*E*)-4-azido-6-(2,4-dimethoxyphenyl)hex-5-enenitrile (3m):



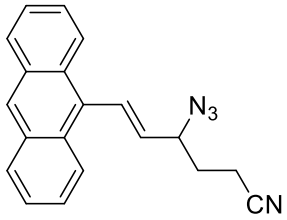
34.8 mg, 64% yield; Light yellow liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.35 (d, *J* = 8.0 Hz, 1H), 6.91 (d, *J* = 16.0 Hz, 1H), 6.48 (d, *J* = 8.5 Hz, 1H), 6.45 (s, 1H), 6.03 – 5.97 (m, 1H), 4.20 – 4.14 (m, 1H), 3.84 (s, 3H), 3.82 (s, 3H), 2.48 – 2.44 (m, 2H), 1.94 – 1.89 (m, 2H). ^{13}C NMR (125 MHz, Chloroform-d) δ 161.2, 158.3, 130.1, 128.2, 122.7, 118.9, 117.3, 104.9, 98.4, 63.9, 55.4, 55.4, 30.6, 14.0. HRMS *m/z* (ESI) calcd. for $C_{14}H_{17}N_4O_2$([M+H]$^+$) 273.1346, found 273.1344.

(*E*)-4-azido-6-(naphthalen-2-yl)hex-5-enenitrile (3n):



38.3 mg, 73% yield; Light yellow solid; mp 105.3-109.5 °C; ^{1}H NMR (500 MHz, Chloroform-*d*) δ1H NMR (500 MHz, Chloroform-d) δ 7.73 (d, *J* = 5.5 Hz, 3H), 7.69 (s, 1H), 7.50 (d, *J* = 8.5 Hz, 1H), 7.39 (d, *J* = 4.0 Hz, 2H), 6.77 (d, *J* = 15.5 Hz, 1H), 6.12 – 6.07 (m, 1H), 4.19 – 4.14 (m, 1H), 2.43 – 2.35 (m, 2H), 1.88 – 1.83 (m, 2H). ^{13}C NMR (125 MHz, Chloroform-d) δ 135.1, 133.4, 133.4, 132.7, 128.4, 128.1, 127.7, 127.3, 126.5, 126.4, 125.0, 123.3, 118.8, 63.1, 30.4, 14.0. HRMS *m/z* (ESI) calcd. for $C_{16}H_{15}N_4$([M+H]$^+$) 263.1291, found 263.1291.

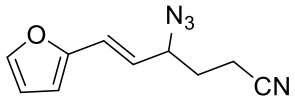
(*E*)-6-(anthracen-9-yl)-4-azidohex-5-enenitrile (3o):



41.8 mg, 67% yield; Light yellow liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.43 (s, 1H), 8.21 (d, *J* = 8.5 Hz, 2H), 8.02 (d, *J* = 8.0 Hz, 2H), 7.53 (s, 1H), 7.49 (d, *J* = 10.5 Hz, 4H), 6.01 – 5.95 (m, 1H), 4.59 – 4.54 (m, 1H), 2.67 – 2.57 (m, 2H), 2.15 – 2.03 (m, 2H). ^{13}C NMR (125 MHz, Chloroform-d) δ 133.7, 131.4, 131.3, 130.4,

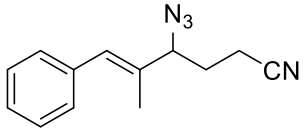
129.3, 128.8, 127.2, 126.0, 125.3, 118.7, 63.4, 30.5, 14.2. HRMS *m/z* (ESI) calcd. for $C_{20}H_{17}N_4([M+H]^+)$ 313.1448, found 313.1449.

(*E*)-4-azido-6-(furan-2-yl)hex-5-enenitrile (3p):



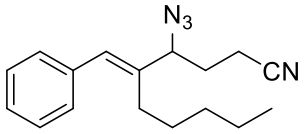
26.7 mg, 66% yield; Gray liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.39 (s, 1H), 6.52 (d, *J* = 18.5 Hz, 1H), 6.38 (d, *J* = 27.5 Hz, 2H), 6.04 – 5.98 (m, 1H), 4.21 – 4.15 (m, *J* = 8.0 Hz, 1H), 2.52 – 2.42 (m, 2H), 1.94 – 1.89 (m, 2H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 150.9, 142.8, 122.9, 122.9, 118.7, 111.6, 110.1, 62.7, 30.4, 13.9. HRMS *m/z* (ESI) calcd. for $C_{10}H_{11}N_4O([M+H]^+)$ 203.0927, found 203.0924.

(*E*)-4-azido-5-methyl-6-phenylhex-5-enenitrile (3q):



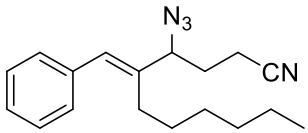
34.8 mg, 77% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.36 (t, *J* = 7.5 Hz, 2H), 7.31 – 7.26 (m, 3H), 6.61 (s, 1H), 4.19 – 4.15 (m, 1H), 2.47 – 2.43 (m, 2H), 1.96 – 1.90 (m, 2H), 1.90 – 1.87 (m, 3H).^{13}C NMR (125 MHz, Chloroform-*d*) δ 136.1 , 133.4 , 130.6 , 129.0 , 128.2 , 127.2 , 118.7 , 68.9 , 28.5 , 14.2 , 13.3. HRMS *m/z* (ESI) calcd. for $C_{13}H_{15}N_4([M+H]^+)$ 227.1291, found 227.1290.

(*E*)-4-azido-5-benzylidenedecanenitrile (3r):



42.8 mg, 76% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.28 (d, *J* = 8.0 Hz, 2H), 7.19 (d, *J* = 8.0 Hz, 3H), 6.52 (s, 1H), 4.07 (t, *J* = 6.5 Hz, 1H), 2.42 (t, *J* = 7.5 Hz, 2H), 2.25 – 2.11 (m, 2H), 1.92 – 1.86 (m, 2H), 1.47 – 1.38 (m, 2H), 1.23 – 1.18 (m, 4H), 0.80 (s, 3H).^{13}C NMR (125 MHz, Chloroform-*d*) δ 138.6, 136.3, 130.2, 128.7, 128.4, 127.2, 118.9, 67.0, 32.0, 29.31, 28.7, 28.4, 22.2, 14.4, 13.9. HRMS *m/z* (ESI) calcd. for $C_{17}H_{23}N_4([M+H]^+)$ 283.1917, found 283.1906.

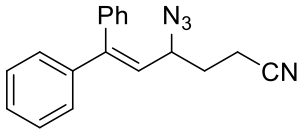
(*E*)-4-azido-5-benzylideneundecanenitrile (3s):



42.0 mg, 74% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.28 (d, *J* = 7.5 Hz, 2H), 7.19 (d, *J* = 8.0 Hz, 3H), 6.52 (s, 1H), 4.07 (t, *J* = 6.5 Hz, 1H), 2.42 (t, *J* = 7.5 Hz, 2H), 2.24 – 2.12 (m, 2H), 1.92 – 1.86 (m, 2H), 1.47 – 1.35 (m, 2H), 1.25-1.18 (m, 6H), 0.80 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 138.6, 136.3,

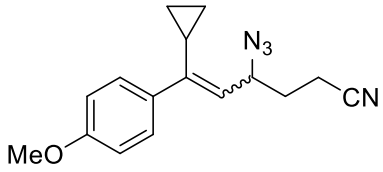
130.2, 128.7, 128.4, 127.2, 118.9, 67.0, 31.4, 29.5, 29.3, 28.9, 28.7, 22.5, 14.4, 14.0. HRMS *m/z* (ESI) calcd. for $C_{18}H_{25}N_4$([M+H]$^+$) 285.1822, found 285.1821.

4-azido-6,6-diphenylhex-5-enenitrile (3t):



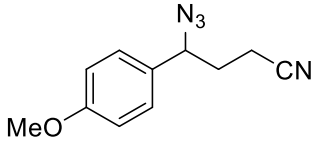
46.7 mg, 81% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.42 (t, *J* = 7.5 Hz, 2H), 7.38 (d, *J* = 7.0 Hz, 1H), 7.29 (d, *J* = 4.5 Hz, 5H), 7.17 (d, *J* = 7.5 Hz, 2H), 6.00 (d, *J* = 10.0 Hz, 1H), 4.18 – 4.12 (m, *J* = 8.0 Hz, 1H), 2.43 – 2.30 (m, 2H), 1.91-1.86 – 5.92 (m, *J* = 7.5 Hz, 2H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 148.1, 140.3, 138.0, 129.5, 128.7, 128.4, 128.3, 128.0, 127.4, 123.6, 118.7, 59.2, 30.8, 13.9. HRMS *m/z* (ESI) calcd. for $C_{18}H_{17}N_4$([M+H]$^+$) 289.1448, found 289.1455.

(*E*)-4-azido-6-cyclopropyl-6-(4-methoxyphenyl)hex-5-enenitrile (3u):



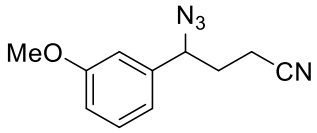
40.6 mg, 72% yield; Reddish liquid; *E*/*Z*=3:1; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.17 (d, *J* = 8.0 Hz, 0.56), 6.92 (d, *J* = 7.5 Hz, 1.44H), 6.81 (d, *J* = 8.0 Hz, 1.44H), 6.76 (d, *J* = 8.0 Hz, 0.56H), 5.41 (d, *J* = 9.5 Hz, 0.28H),5.29 (d, *J* = 9.5 Hz, 0.72H), 4.82 – 4.76 (m, 0.28H), 3.87 – 3.82 (m, 0.72H),3.72 (s, 3H), 2.44 – 2.36 (m, 0.56H), 2.27 – 2.17 (m, 1.44H), 1.85 – 1.76 (m, 0.56H), 1.74 – 1.66 (m, 1.44H), 1.65 – 1.62 (m, 0.28H), 1.60 – 1.53 (m, 0.72H), 0.87 – 0.79 (m, 0.56H), 0.65 – 0.61 (m, 1.44H), 0.42 – 0.30 (m, 2H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 159.0, 158.9, 150.4, 148.0, 132.5, 129.7, 129.1, 128.7, 124.5, 120.5, 118.9, 118.8, 113.6, 113.2, 58.8, 57.7, 55.1, 55.1, 30.7, 30.6, 18.5, 14.0, 13.7, 11.8, 7.3, 6.7, 5.9, 5.3. HRMS *m/z* (ESI) calcd. for $C_{16}H_{19}N_4O$([M+H]$^+$) 283.1553, found 283.1548.

4-azido-4-(4-methoxyphenyl)butanenitrile (3v):



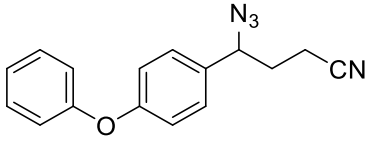
30.3 mg, 70% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.24 (d, *J* = 8.5 Hz, 2H), 6.94 (d, *J* = 8.5 Hz, 2H), 4.59 – 4. 55 (m, 1H), 3.82 (s, 3H), 2.47 – 2.33 (m, 2H), 2.11 – 1.97 (m, 2H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 159.9 , 129.5 , 128.1 , 118.7 , 114.5 , 63.9 , 55.3 , 31.9 , 14.3 , 62.7 , 30.2 , 13.8. HRMS *m/z* (ESI) calcd. for $C_{11}H_{13}N_4O$ ([M+H]$^+$) 217.1084, found 217.1086.

4-azido-4-(3-methoxyphenyl)butanenitrile (3w):



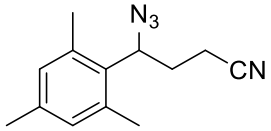
26.4 mg, 61% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.28 – 7.24 (m, 1H), 6.83 (d, *J* = 6.5 Hz, 2H), 6.78 (s, 1H), 4.53 (t, *J* = 7.0 Hz, 1H), 3.76 (s, 3H), 2.44 – 2.37 (m, 1H), 2.33 – 2.25 (m, 1H), 2.04 – 1.92 (m, 2H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 160.1, 139.2, 130.3, 119.0, 118.7, 114.2, 112.5, 64.2, 55.3, 31.9, 14.2. HRMS *m/z* (ESI) calcd. for $C_{11}H_{13}N_4O$ ([M+H]$^+$) 217.1084, found 217.1087.

4-azido-4-(4-phenoxyphenyl)butanenitrile (3x):



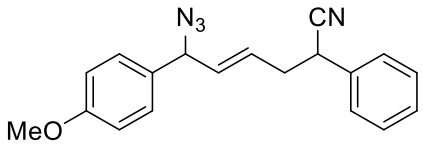
45.1 mg, 81% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.30 – 7.27 (m, 2H), 7.20 (d, *J* = 6.0 Hz, 2H), 7.07 (t, *J* = 7.5 Hz, 1H), 6.97 – 6.93 (m, 4H), 4.53 (t, *J* = 7.0 Hz, 1H), 2.44 – 2.27 (m, 2H), 2.06 – 1.89 (m, 2H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 158.0, 156.4, 132.1, 129.9, 128.3, 123.8, 119.4, 118.9, 118.6, 63.8, 31.9, 14.3. HRMS *m/z* (ESI) calcd. for $C_{16}H_{15}N_4O$([M+H]$^+$) 279.1240, found 279.1240.

4-azido-4-mesitylbutanenitrile (3y):



25.6 mg, 56% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 6.81 (s, 2H), 5.14 – 5.09 (m, 1H), 2.47 – 2.37 (m, 2H), 2.34 (s, 6H), 2.19 (s, 3H), 2.16 – 2.11 (m, 1H), 1.90 – 1.82 (m, 1H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 138.0, 136.4, 130.6, 118.9, 60.4, 29.4, 20.8, 20.6, 15.0. HRMS *m/z* (ESI) calcd. for $C_{13}H_{17}N_4$([M+H]$^+$) 229.1448, found 229.1456.

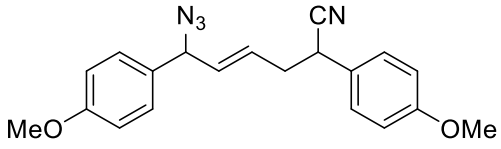
(*E*)-6-azido-6-(4-methoxyphenyl)-2-phenylhex-4-enenitrile (5a):



45.8 mg, 72% yield; Light yellow liquid; dr= 3:2; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.35 – 7.24 (m, 7H), 6.79 (d, *J* = 8.5 Hz, 2H), 6.59 – 6.49 (m, 1H), 5.90 – 5.83 (m, 1H), 4.24 (d, *J* = 12.0 Hz, 0.6H), 3.99 – 3.95 (m, 0.6H), 3.92 – 3.86 (m, 0.4H), 3.83 (d, *J* = 8.0 Hz, 0.4H), 3.73 (s, 3H), 2.24 – 2.17 (m, 0.4H), 2.09 – 2.03 (m, 0.6H), 2.00 – 1.91 (m, 1H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 160.0, 159.9, 135.0, 135.0, 134.6, 134.5, 129.3, 129.3, 128.5, 128.3, 128.1, 128.0, 128.0, 127.9, 127.5,

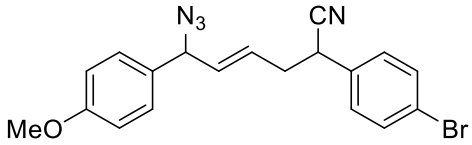
127.2, 122.4, 122.2, 120.4, 120.0, 114.1, 62.5, 61.9, 55.3, 41.0, 40.2, 34.3, 33.8. HRMS *m/z* (ESI) calcd. for $C_{19}H_{19}N_4O([M+H]^+)$ 319.1553, found 319.1549.

(*E*)-6-azido-2,6-bis(4-methoxyphenyl)hex-4-enenitrile (5b):



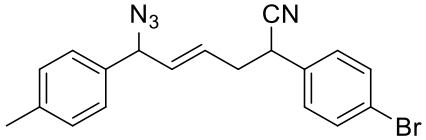
47.3 mg, 68% yield; Light yellow liquid; dr= 1:1; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.33 (d, *J* = 8.5 Hz, 2H), 7.24 (d, *J* = 6.5 Hz, 2H), 6.93 – 6.86 (m, 4H), 6.65 – 6.55 (m, 1H), 5.97 – 5.90 (m, 1H), 4.31 – 4.25 (m, 0.5H), 4.00 – 3.96 (m, 0.5H), 3.93 (t, *J* = 8.0 Hz, 0.5H),3.86 (t, *J* = 8.0 Hz, 0.5H), 3.81 (s, 6H), 2.29 – 2.22 (m, 0.5H), 2.15 – 2.08 (m, 0.5H), 2.04 – 1.96 (m, 1H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 160.0, 159.9, 159.6, 159.5, 134.8, 134.6, 128.7, 128.4, 128.1, 128.0, 128.0, 126.9, 126.3, 122.5, 122.3, 120.6, 120.3, 114.7, 114.6, 114.1, 62.5, 61.8, 55.3, 55.3, 41.0, 40.2, 33.5, 33.0. HRMS *m/z* (ESI) calcd. for $C_{20}H_{21}N_4O_2$ ($[M+H]^+$) 349.1659, found 349.1658.

(*E*)-6-azido-2-(4-bromophenyl)-6-(4-methoxyphenyl)hex-4-enenitrile (5c):



51.5 mg, 65% yield; Light yellow liquid; dr= 7:3;^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.54 (d, *J* = 9.0 Hz, 2H), 7.33 (d, *J* = 7.0 Hz, 2H), 7.22 (d, *J* = 8.0 Hz, 2H), 6.88 (d, *J* = 8.0 Hz, 2H), 6.67 – 6.58 (m, 1H), 5.97 – 5.89 (m, 1H), 4.32 (t, *J* = 9.0 Hz, 0.7H), 4.05 – 4.01 (m, 0.7H), 3.98 – 3.92 (m, 0.3H), 3.89 (t, *J* = 8.0 Hz, 0.3H), 3.82 (s, 3H), 2.31 – 2.24 (m, 0.3H), 2.14 – 2.08 (m, 0.7H), 2.05 – 1.95 (m, 1H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 160.0, 135.3, 134.8, 134.1, 132.5, 132.5, 129.2, 128.9, 128.1, 128.1, 127.9, 122.7, 122.5, 122.2, 121.9, 119.5, 114.2, 62.5, 61.7, 55.3, 41.0, 40.1, 33.9, 33.4. HRMS *m/z* (ESI) calcd. for $C_{19}H_{18}BrN_4O([M+H]^+)$ 397.0659, found 397.0655.

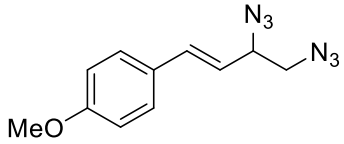
(*E*)-6-azido-2-(4-bromophenyl)-6-(p-tolyl)hex-4-enenitrile (5d):



41.0 mg, 54% yield; Light yellow liquid; dr= 11:9; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.45 (t, *J* = 9.5 Hz, 2H), 7.23 – 7.19 (m, 2H), 7.17 – 7.12 (m, 2H), 7.07 (d, *J* = 8.0 Hz, 2H), 6.61 – 6.52 (m, 1H), 5.97 – 5.90 (m, 1H), 4.25 (t, *J* = 10.0 Hz, 0.55H), 3.96 – 3.93 (m, 0.55H), 3.90 – 3.84 (m, 0.45H), 3.80(t, *J* = 7.5

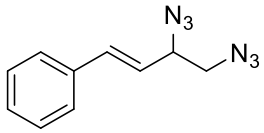
Hz,0.45H), 2.27 (s, 3H), 2.22 – 2.16 (m, 0.45H), 2.06 – 2.00 (m, 0.55H), 1.97 – 1.87 (m, 1H).^{13}C NMR (125 MHz, Chloroform-*d*) δ 138.8, 138.7, 135.6, 135.2, 134.0, 133.5, 132.5, 132.4, 129.4, 129.2, 128.9, 126.7, 126.7, 123.5, 123.2, 122.7, 122.4, 119.8, 119.5, 62.3, 61.6, 40.8, 40.0, 33.9, 33.3, 21.2. HRMS *m/z* (ESI) calcd. for $C_{19}H_{18}BrN_4$([M+H]$^+$) 381.0709, found 381.0707.

(*E*)-1-(3,4-diazidobut-1-en-1-yl)-4-methoxybenzene (4a):



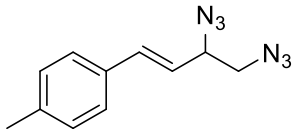
46.4 mg, 95% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.35 (d, *J* = 8.5 Hz, 2H), 6.87 (d, *J* = 8.5 Hz, 2H), 6.66 (d, *J* = 15.5 Hz, 1H), 6.00 – 5.95 (m, 1H), 4.25 – 4.20 (m, 1H), 3.81 (s, 3H), 3.37 (t, *J* = 6.0 Hz, 2H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 159.9, 135.1, 128.1, 120.5, 114.1, 64.1, 55.3, 54.6. HRMS *m/z* (ESI) calcd. for $C_{11}H_{13}N_6O$ ([M+H]$^+$) 245.1145, found 245.1145.

(*E*)-(3,4-diazidobut-1-en-1-yl)benzene (4b):



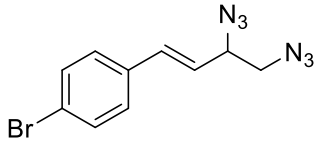
39.0 mg, 91% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.34 (d, *J* = 7.0 Hz, 2H), 7.28 (t, *J* = 7.5 Hz, 2H), 7.25 – 7.21 (m, 1H), 6.66 (d, *J* = 16.0 Hz, 1H), 6.08 – 6.02 (m, 1H), 4.22 – 4.16 (m, 1H), 3.36 – 3.28 (m, 2H).^{13}C NMR (125 MHz, Chloroform-*d*) δ 135.5 , 135.3 , 128.7 , 128.6 , 126.8 , 122.9 , 63.9 , 54.5. HRMS *m/z* (ESI) calcd. for $C_{10}H_{11}N_6$([M+H]$^+$) 215.1040, found 215.1041.

(*E*)-1-(3,4-diazidobut-1-en-1-yl)-4-methylbenzene (4c):



42.4 mg, 93% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.22 (d, *J* = 7.5 Hz, 2H), 7.07 (d, *J* = 7.5 Hz, 2H), 6.61 (d, *J* = 16.0 Hz, 1H), 6.01 – 5.96 (m, 1H), 4.18 – 4.13 (m, 1H), 3.29 (t, *J* = 6.0 Hz, 2H), 2.27 (s, 3H).^{13}C NMR (125 MHz, Chloroform-*d*) δ 138.6 , 135.5 , 132.5 , 129.4 , 126.7 , 121.8 , 64.0 , 54.5 , 21.2. HRMS *m/z* (ESI) calcd for $C_{11}H_{13}N_6$ ([M+H]$^+$) 229.1196, found 229.1197.

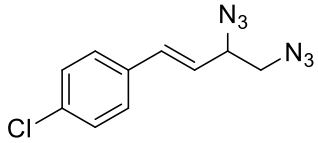
(*E*)-1-bromo-4-(3,4-diazidobut-1-en-1-yl)benzene (4d):



50.2 mg, 86% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.38 (d, *J* = 8.5 Hz, 2H), 7.18 (d, *J* = 8.5 Hz, 2H), 6.57 (d, *J* = 16.0 Hz, 1H), 6.05 – 5.99 (m, 1H), 4.18

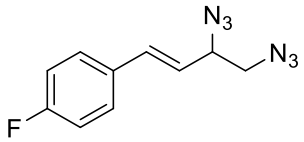
– 4.12 (m, 1H), 3.34 – 3.26 (m, 2H).^{13}C NMR (125 MHz, Chloroform-*d*) δ 134.2 , 134.1 , 131.8 , 128.2 , 123.8 , 122.4 , 63.6 , 54.3. HRMS *m/z* (ESI) calcd. for $C_{10}H_{10}BrN_6$([M+H]$^+$)293.0145, found 293.0145.

(*E*)-1-chloro-4-(3,4-diazidobut-1-en-1-yl)benzene (4e):



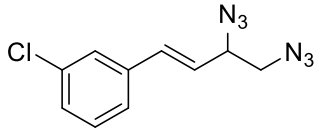
41.2 mg, 83% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.36 – 7.29 (m, 4H), 6.68 (d, *J* = 16.0 Hz, 1H), 6.12 – 6.07 (m, 1H), 4.28 – 4.21 (m, 1H), 3.42 – 3.36 (m, 2H).^{13}C NMR (126 MHz, Chloroform-*d*) δ 134.3 , 134.1 , 133.8 , 128.9 , 128.0 , 123.7 , 63.7 , 54.4. HRMS *m/z* (ESI) calcd. for $C_{10}H_{10}ClN_6$ ([M+H]$^+$) 249.0650, found 249.0652.

(*E*)-1-(3,4-diazidobut-1-en-1-yl)-4-fluorobenzene (4f):



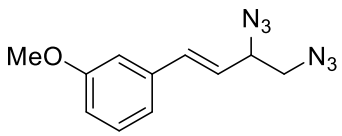
41.8 mg, 90% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.31 (t, *J* = 7.0 Hz, 2H), 6.96 (t, *J* = 8.5 Hz, 2H), 6.62 (d, *J* = 15.5 Hz, 1H), 5.97 (m, 1H), 4.19 – 4.15 (m, 1H), 3.36 – 3.28 (m, 2H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 163.9, 161.9, 134.3, 131.6, 131.5, 128.5, 128.4, 122.8, 122.7, 115.8, 115.6, 77.3, 63.8, 54.5. ^{19}F NMR (471 MHz, Chloroform-d) δ -112.61. HRMS *m/z* (ESI) calcd. for $C_{10}H_{10}FN_6$([M+H]$^+$) 233.0945, found 233.0945.

(*E*)-1-chloro-3-(3,4-diazidobut-1-en-1-yl)benzene (4g):



38.7 mg, 78% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.39 (s, 1H), 7.27 (s, 3H), 6.67 (d, *J* = 16.0 Hz, 1H), 6.16 – 6.10 (m, 1H), 4.27 – 4.22 (m, 1H), 3.43 – 3.36 (m, 2H).^{13}C NMR (125 MHz, Chloroform-*d*) δ 137.2 , 134.7 , 134.0 , 130.0 , 128.5 , 126.6 , 125.0 , 124.6 , 63.6 , 54.4. HRMS *m/z* (ESI) calcd. for $C_{10}H_{10}ClN_6$([M+H]$^+$) 249.0650, found 249.0649.

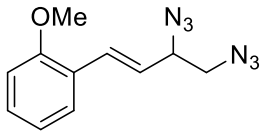
(*E*)-1-(3,4-diazidobut-1-en-1-yl)-3-methoxybenzene (4h):



40.0 mg, 82% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.19 (t, *J* = 7.5 Hz, 1H), 6.93 (d, *J* = 7.5 Hz, 1H), 6.86 (s, 1H), 6.78 (d, *J* = 8.5 Hz, 1H), 6.62 (d, *J* = 16.0 Hz, 1H), 6.06 – 6.01 (m, 1H), 4.20 – 4.14 (m, 1H), 3.75 (s, 3H), 3.35 – 3.27 (m,

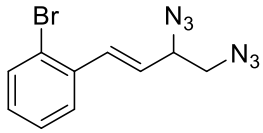
2H).^{13}C NMR (125 MHz, Chloroform-*d*) δ 159.8, 136.8, 135.4, 129.7, 123.3, 119.4, 114.2, 112.1, 63.8, 55.2, 54.5. HRMS *m/z* (ESI) calcd. for $C_{11}H_{13}N_6O([M+H]^+)$ 245.1145, found 245.1151.

(*E*)-1-(3,4-diazidobut-1-en-1-yl)-2-methoxybenzene (4i):



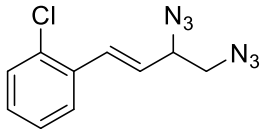
43.5 mg, 89% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.44 (d, *J* = 9.5 Hz, 1H), 7.29 – 7.26 (m, 1H), 7.04 (d, *J* = 16.0 Hz, 1H), 6.94 (t, *J* = 7.5 Hz, 1H), 6.89 (d, *J* = 8.0 Hz, 1H), 6.19 – 6.13 (m, 1H), 4.28 – 4.24 (m, 1H), 3.86 (s, 3H), 3.42 – 3.36 (m, 2H).^{13}C NMR (125 MHz, Chloroform-*d*) δ 157.1 , 130.7 , 129.7 , 127.3 , 124.3 , 123.4 , 120.7 , 111.0 , 64.5 , 55. 5 , 54.7. HRMS *m/z* (ESI) calcd. for $C_{11}H_{13}N_6O$ ($[M+H]^+$) 245.1145, found 245.1145.

(*E*)-1-chloro-3-(3,4-diazidobut-1-en-1-yl)benzene (4j):



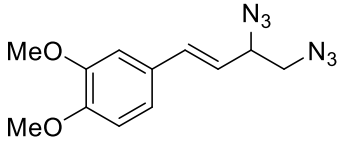
53.7 mg, 92% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.57 (d, *J* = 8.0 Hz, 1H), 7.52 (d, *J* = 8.0 Hz, 1H), 7.29 (t, *J* = 7.5 Hz, 1H), 7.16 (t, *J* = 8.0 Hz, 1H), 7.07 (d, *J* = 15.5 Hz, 1H), 5.10 – 5.04 (m, 1H), 4.33 – 4.28 (m, 1H), 3.46 – 3.37 (m, 2H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 135.42, 134.30, 133.04, 129.79, 127.61, 127.27, 125.95, 123.85, 63.53, 54.40. HRMS *m/z* (ESI) calcd. for $C_{10}H_{10}BrN_6([M+H]^+)$ 293.0145, found 293.0151.

(*E*)-1-chloro-2-(3,4-diazidobut-1-en-1-yl)benzene (4k):



42.7 mg, 86% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.48 – 7.44 (m, 1H), 7.33 – 7.27 (m, 1H), 7.18 – 7.15 (m, 2H), 7.04 (d, *J* = 16.0 Hz, 1H), 6.01 – 6.01 (m, 1H), 4.26 – 4.20 (m, 1H), 3.39 – 3.30 (m, 2H).^{13}C NMR (125 MHz, Chloroform-*d*) δ 133.6 , 133.4 , 131.7 , 129.8 , 129.6 , 127.1 , 127.0 , 125. 8 , 63.7 , 54.4. HRMS *m/z* (ESI) calcd for $C_{10}H_{10}ClN_6$ ($[M+H]^+$) 249.0650, found 249.0649.

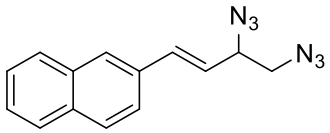
(*E*)-4-(3,4-diazidobut-1-en-1-yl)-1,2-dimethoxybenzene (4l):



47.7 mg, 87% yield; Light yellow solid; mp 74.5-79.2 °C; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 6.87 (t, *J* = 4.0 Hz, 2H), 6.74 (d, *J* = 6.5 Hz, 1H), 6.57 (d, *J* = 15.5 Hz, 1H),

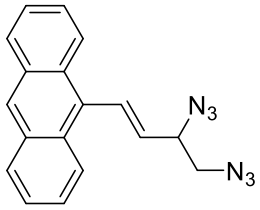
5.93 – 5.87 (m, 1H), 4.17 – 4.12 (m, 1H), 3.82 (s, 3H), 3.79 (s, 3H), 3.33 – 3.24 (m, 2H).^{13}C NMR (125 MHz, Chloroform-*d*) δ 149.5, 149.0, 135.1, 128.2, 120.6, 120.2, 110.9, 108.8, 63.9, 55.7, 55.7, 54.4. HRMS *m/z* (ESI) calcd. for $C_{12}H_{15}N_6O_2([M+H]^+)$ 275.1251, found 275.1253.

(*E*)-2-(3,4-diazidobut-1-en-1-yl)naphthalene (4m):



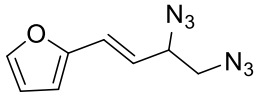
47.0 mg, 89% yield; white solid; mp 75.2-89.6 °C; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.70 (d, *J* = 7.0 Hz, 3H), 7.66 (s, 1H), 7.48 (d, *J* = 8.5 Hz, 1H), 7.38 (d, *J* = 4.0 Hz, 2H), 6.76 (d, *J* = 15.5 Hz, 1H), 6.15 – 6.09 (m, 1H), 4.20 – 4.15 (m, 1H), 3.33 – 3.26 (m, 2H).^{13}C NMR (125 MHz, Chloroform-*d*) δ 135.5, 133.4, 132.7, 128.4, 128.1, 127.7, 127.3, 126.5, 126.4, 123.3, 123.2, 63.9, 54.5. HRMS *m/z* (ESI) calcd. for $C_{14}H_{13}N_6([M+H]^+)$ 265.1196, found 265.1185.

(*E*)-9-(3,4-diazidobut-1-en-1-yl)anthracene (4n):



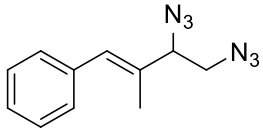
54.0 mg, 86% yield; Reddish liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.30 (s, 1H), 8.11 (d, *J* = 8.0 Hz, 2H), 7.92 (d, *J* = 8.0 Hz, 2H), 7.46 – 7.39 (m, 4H), 7.32 (d, *J* = 16.0 Hz, 1H), 5.87 – 5.82 (m, 1H), 4.43 – 4.38 (m, 1H), 3.44 (t, *J* = 4.5 Hz, 2H).^{13}C NMR (125 MHz, Chloroform-*d*) δ 131.8, 131.6, 131.2, 130.5, 129.1, 128.7, 127.1, 125.8, 125.3, 125.1, 64.0, 54.4. HRMS *m/z* (ESI) calcd. for $C_{18}H_{15}N_6([M+H]^+)$ 315.1353, found 315.1359.

(*E*)-2-(3,4-diazidobut-1-en-1-yl)furan (4o):



34.7 mg, 85% yield; Grey liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.39 (s, 1H), 6.53 (d, *J* = 15.5 Hz, 1H), 6.40 (s, 1H), 6.35 (s, 1H), 6.08 – 6.02 (m, 1H), 4.24 – 4.19 (m, 1H), 3.41 – 3.36 (m, 2H).^{13}C NMR (125 MHz, Chloroform-*d*) δ 151.0, 142.8, 123.2, 121.3, 111.5, 110.1, 63.6, 54.5. HRMS *m/z* (ESI) calcd. for $C_8H_9N_6O([M+H]^+)$ 205.0832, found 205.0835.

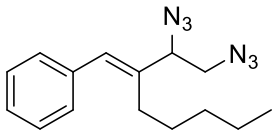
(*E*)-(3,4-diazido-2-methylbut-1-en-1-yl)benzene (4p):



42.0 mg, 92% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.36 (t, *J* = 7.5 Hz, 2H), 7.32 – 7.26 (m, 3H), 6.63 (s, 1H), 4.25 – 4.18 (m, 1H), 3.41 – 3.39 (m, 2H), 1.90 (d,

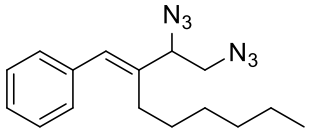
J = 1.5 Hz, 3H).^{13}C NMR (125 MHz, Chloroform-*d*) δ 136.2 , 132.4 , 131.0 , 129.0 , 128.3 , 127.3 , 69.5 , 53.0 , 13.9. HRMS *m/z* (ESI) calcd. for $C_{11}H_{13}N_6$ ([M+H]$^+$) 229.1196, found 229.1197.

(*E*)-(2-(1,2-diazidoethyl)hept-1-en-1-yl)benzene (4q):



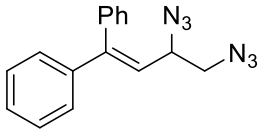
50.6 mg, 89% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.28 (t, *J* = 8.0 Hz, 2H), 7.19 (t, *J* = 8.0 Hz, 3H), 6.54 (s, 1H), 4.11 (t, *J* = 6.5 Hz, 1H), 3.33 (d, *J* = 6.5 Hz, 2H), 2.27 – 2.06 (m, 2H), 1.44 – 1.41 (m, 2H), 1.23 – 1.20 (m, 4H), 0.80 (s, 3H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 137.5, 136.4, 130.3, 128.6, 128.3, 127.2, 67.4, 53.9, 31.9, 29.2, 28.3, 22.2, 13.9. HRMS *m/z* (ESI) calcd. for $C_{15}H_{21}N_6$([M+H]$^+$) 285.1822, found 285.1826.

(E)-(2-(1,2-diazidoethyl)oct-1-en-1-yl)benzene (4r):



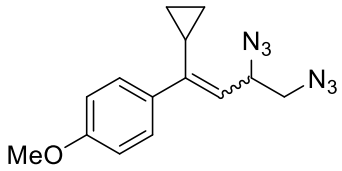
53.6 mg, 90% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.35 (t, *J* = 7.5 Hz, 2H), 7.26 (t, *J* = 7.5 Hz, 3H), 6.62 (s, 1H), 4.19 (t, *J* = 6.5 Hz, 1H), 3.41 (d, *J* = 6.5 Hz, 2H), 2.35 – 2.14 (m, 2H), 1.53 – 1.45 (m, 2H), 1.32 – 1.23 (m, 6H), 0.87 (s, 3H). ^{13}C NMR (125 MHz, Chloroform-d) δ 137.5, 136.4, 130.4, 128.7, 128.7, 128.3, 127.2, 67.4, 53.9, 31.4, 29.4, 29.2, 28.6, 22.5, 14.0. HRMS m/z (ESI) calcd. for $C_{16}H_{23}N_6$([M+H]$^+$) 299.1979, found 299.1984.

(3,4-diazidobut-1-ene-1,1-diyl)dibenzene (4s):



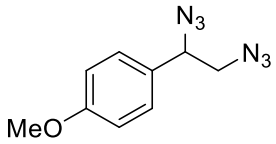
52.8 mg, 91% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.32 (m, 3H), 7.21 (m, 5H), 7.11 (d, *J* = 7.5 Hz, 2H), 5.97 (d, *J* = 10.0 Hz, 1H), 4.17 – 4.12 (m, 1H), 3.26 (d, *J* = 6.0 Hz, 2H). ^{13}C NMR (125 MHz, Chloroform-*d*) δ 148.77, 140.41, 138.13, 129.48, 128.68, 128.41, 128.33, 128.03, 127.48, 121.76, 60.11, 54.56.HRMS *m/z* (ESI) calcd. for $C_{16}H_{15}N_6$([M+H]$^+$) 291.1353, found 291.1346.

(*E*)-1-(3,4-diazido-1-cyclopropylbut-1-en-1-yl)-4-methoxybenzene (4t):



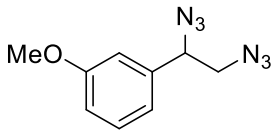
46.0 mg, 81% yield; Colorless liquid; *E*/*Z*=6:1; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.18 (d, *J* = 5.5 Hz, 0.3H),6.94 (d, *J* = 7.5 Hz, 1.7H), 6.82 (d, *J* = 7.5 Hz, 1.7H), 6.78 (d, *J* = 10.0 Hz, 0.3H), 5.47 (d, *J* = 9.5 Hz, 0.15H),5.36 (d, *J* = 9.5 Hz, 0.85H), 4.84 – 4.79 (m, 0.15H),3.97 – 3.92 (m, 0.85H), 3.75 (s, 3.51H), 3.31 (t, *J* = 4.0 Hz, 0.3H), 3.13 (t, *J* = 3.5 Hz, 1.7H), 1.73 – 1.69 (m, 0.15H), 1.62 – 1.55 (m, 0.85H), 0.85 – 0.80 (m, 0.3H), 0.69 – 0.62 (m, 1.7H), 0.44 – 0.33 (m, 2H).^{13}C NMR (125 MHz, Chloroform-*d*) δ 159.0, 151.2, 129.8, 129.4, 128.8, 122.7, 118.7, 113.8, 113.3, 59.8, 58.9, 55.2, 54.6, 18.7, 12.0, 6.8, 6.0, 5.5. HRMS *m/z* (ESI) calcd. for $C_{14}H_{17}N_6O([M+H]^+)$ 285.1458, found 285.1455.

1-(1,2-diazidoethyl)-4-methoxybenzene (4u):



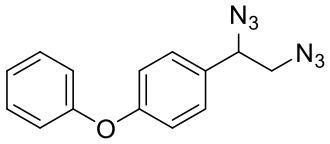
37.5 mg, 86% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.18 (d, *J* = 8.5 Hz, 2H), 6.86 (d, *J* = 8.5 Hz, 2H), 4.56 – 4.53 (m, 1H), 3.74 (s, 3H), 3.43 – 3.31 (m, 2H).^{13}C NMR (125 MHz, Chloroform-*d*) δ 160.0 , 129.80 , 128.2 , 114.4 , 65.0 , 55.8 , 55.3 . HRMS *m/z* (ESI) calcd. for $C_9H_{11}N_6O([M+H]^+)$ 219.0989, found 219.0989.

1-(1,2-diazidoethyl)-3-methoxybenzene (4v):



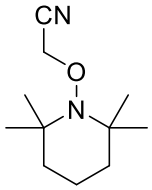
27.5 mg, 63% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.25 (t, *J* = 8.0 Hz, 1H), 6.84 (d, *J* = 8.0 Hz, 2H), 6.80 (s, 1H), 4.59 – 4.54 (m, 1H), 3.76 (s, 3H), 3.45 – 3.34 (m, 2H).^{13}C NMR (125 MHz, Chloroform-*d*) δ 160.1, 137.8, 130.2, 119.1, 114.3, 112.6, 65.5, 55.9, 55.3. HRMS *m/z* (ESI) calcd. for $C_9H_{11}N_6O([M+H]^+)$ 219.0989, found 219.0981.

1-(1,2-diazidoethyl)-4-phenoxybenzene (4w):



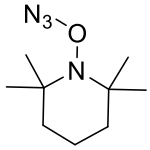
45.9 mg, 82% yield; Colorless liquid; ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.28 (t, *J* = 8.0 Hz, 2H), 7.21 (d, *J* = 8.0 Hz, 2H), 7.07 (t, *J* = 7.5 Hz, 1H), 6.95 (d, *J* = 7.0 Hz, 4H), 4.59 – 4.56 (m, 1H), 3.45 – 3.34 (m, 2H).^{13}C NMR (125 MHz, Chloroform-*d*) δ 158.1, 156.5, 130.8, 129.9, 128.4, 123.8, 119.4, 118.9, 65.0, 55.9. HRMS *m/z* (ESI) calcd. for $C_{14}H_{13}N_6O([M+H]^+)$ 281.1145, found 281.1155.

2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)acetonitrile:



Colorless liquid; ^{1}H NMR (500 MHz, $CDCl_3$) δ1H NMR (500 MHz, Chloroform-d) δ 4.45 (s, 2H), 1.51 – 1.45 (m, m 1H), 1.38 (d, *J* = 16.5 Hz, 4H), 1.28 – 1.24 (m, 1H), 1.13 (s, 6H), 1.03 (s, 6H).^{13}C NMR (125 MHz, $CDCl_3$) δ 115.9, 62.5, 60.2, 39.5, 32.9, 19.8, 16.8. LRMS (EI, 70eV) *m/z* (%): 196 (4), 181 (75), 156 (100), 109 (52); HRMS *m/z* (ESI) calcd for $C_{11}H_{21}N_2O([M+H]^+)$ 197.1648, found 197.1645.

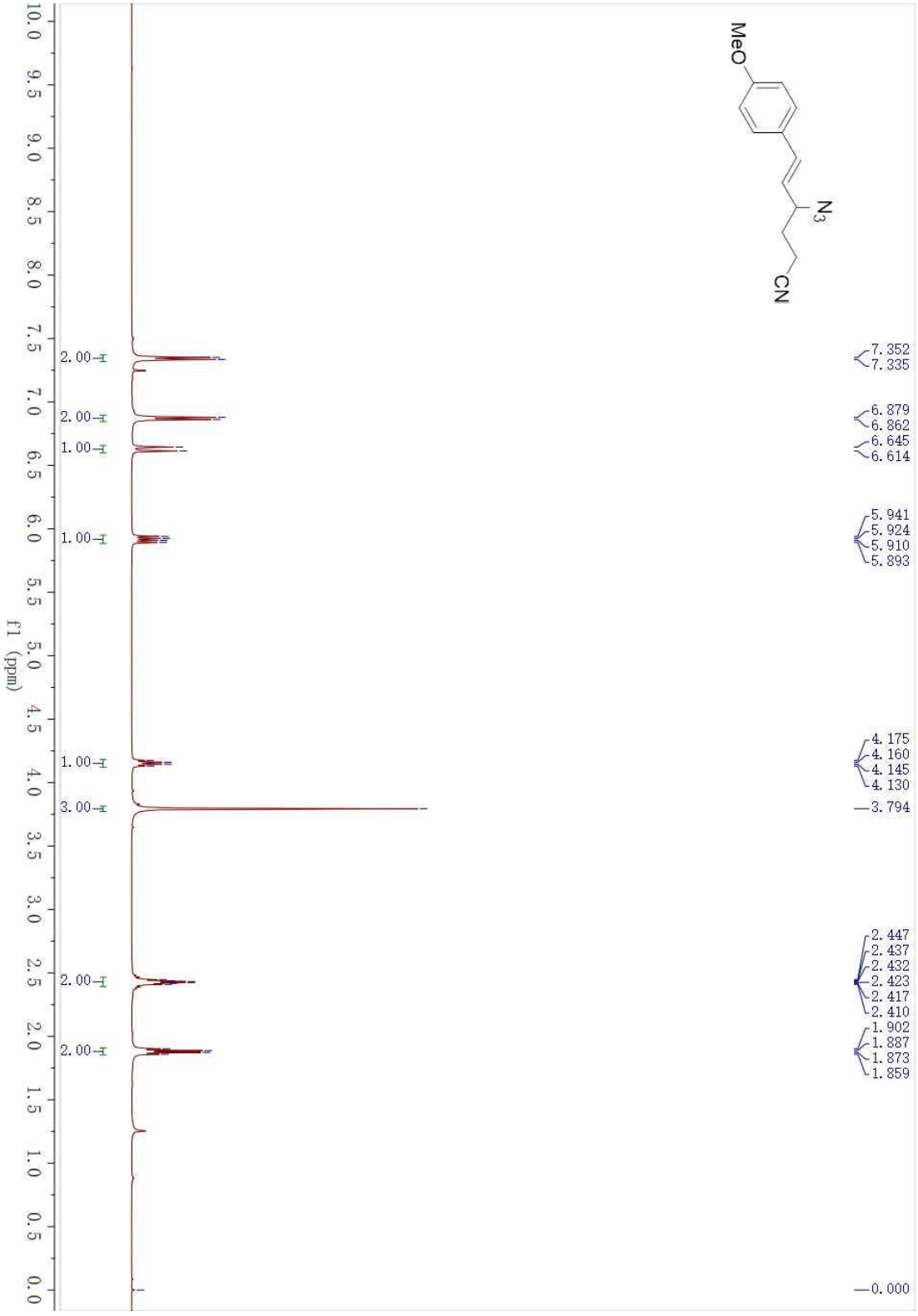
1-(azidooxy)-2,2,6,6-tetramethylpiperidine:

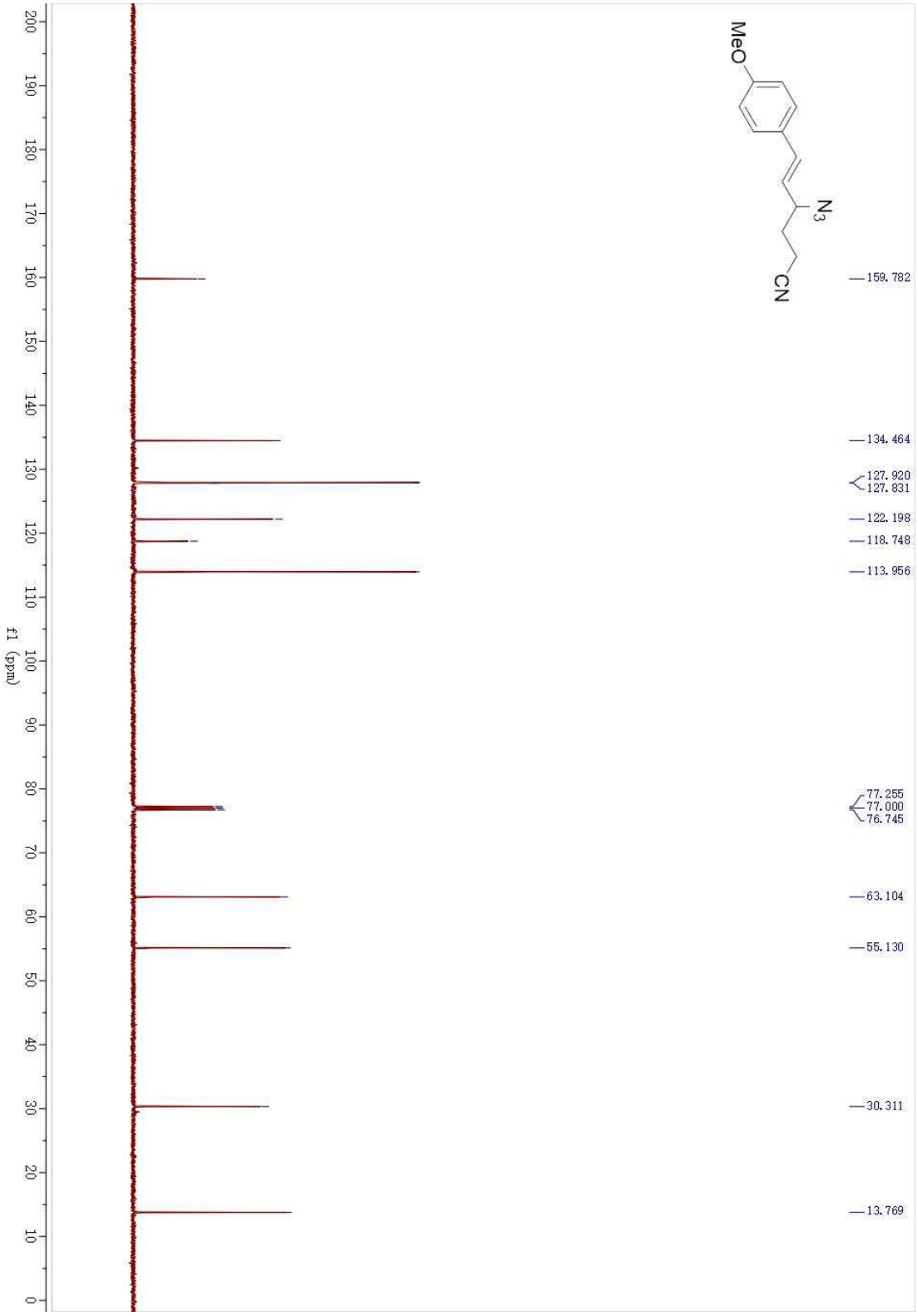


Colorless liquid; LRMS (EI, 70eV) *m/z* (%): 198 (5), 142 (84), 133 (100), 87 (73); HRMS *m/z* (ESI) calcd for $C_9H_{19}N_4O([M+H]^+)$ 199.1553, found 199.1559.

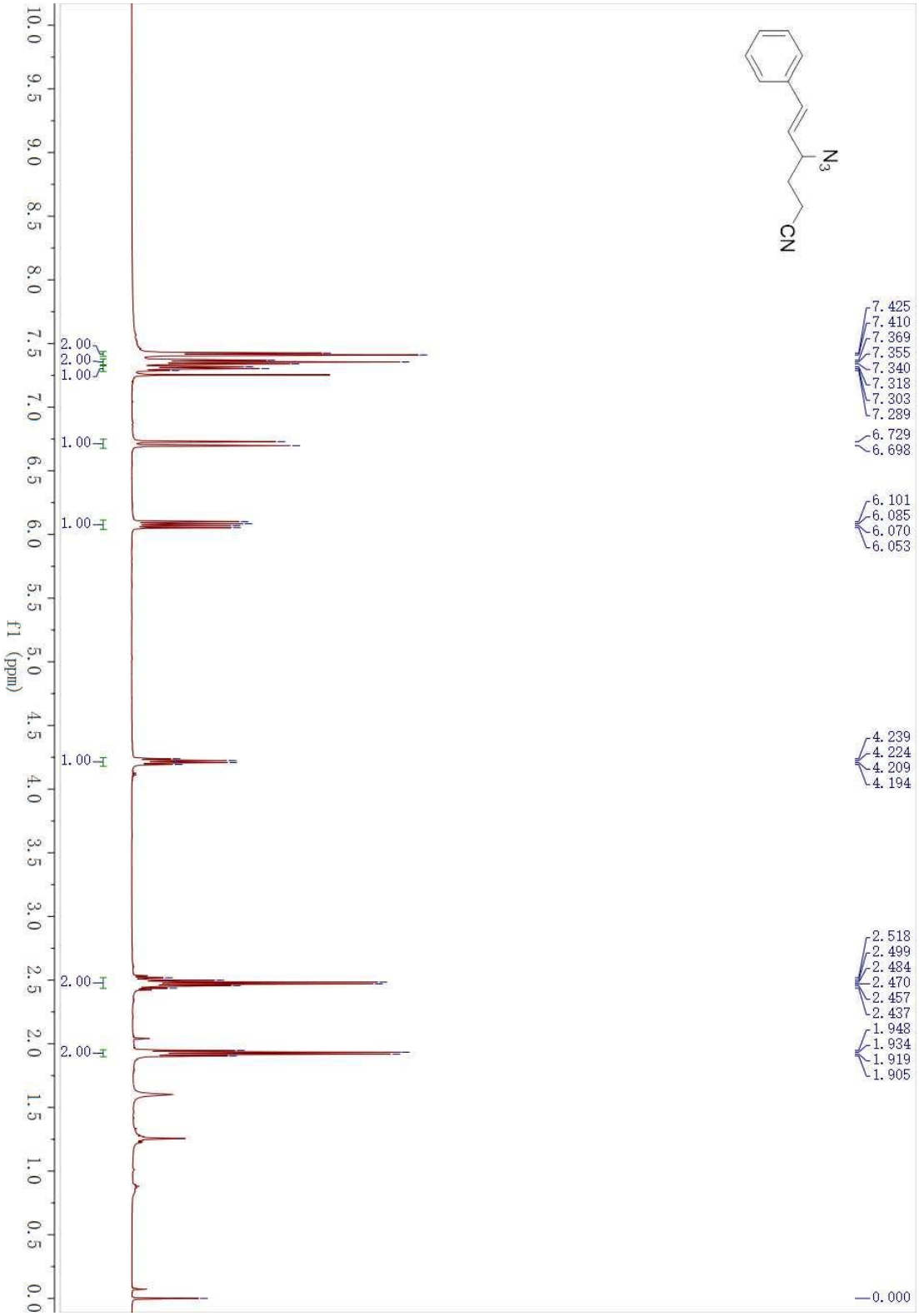
(C) Spectra

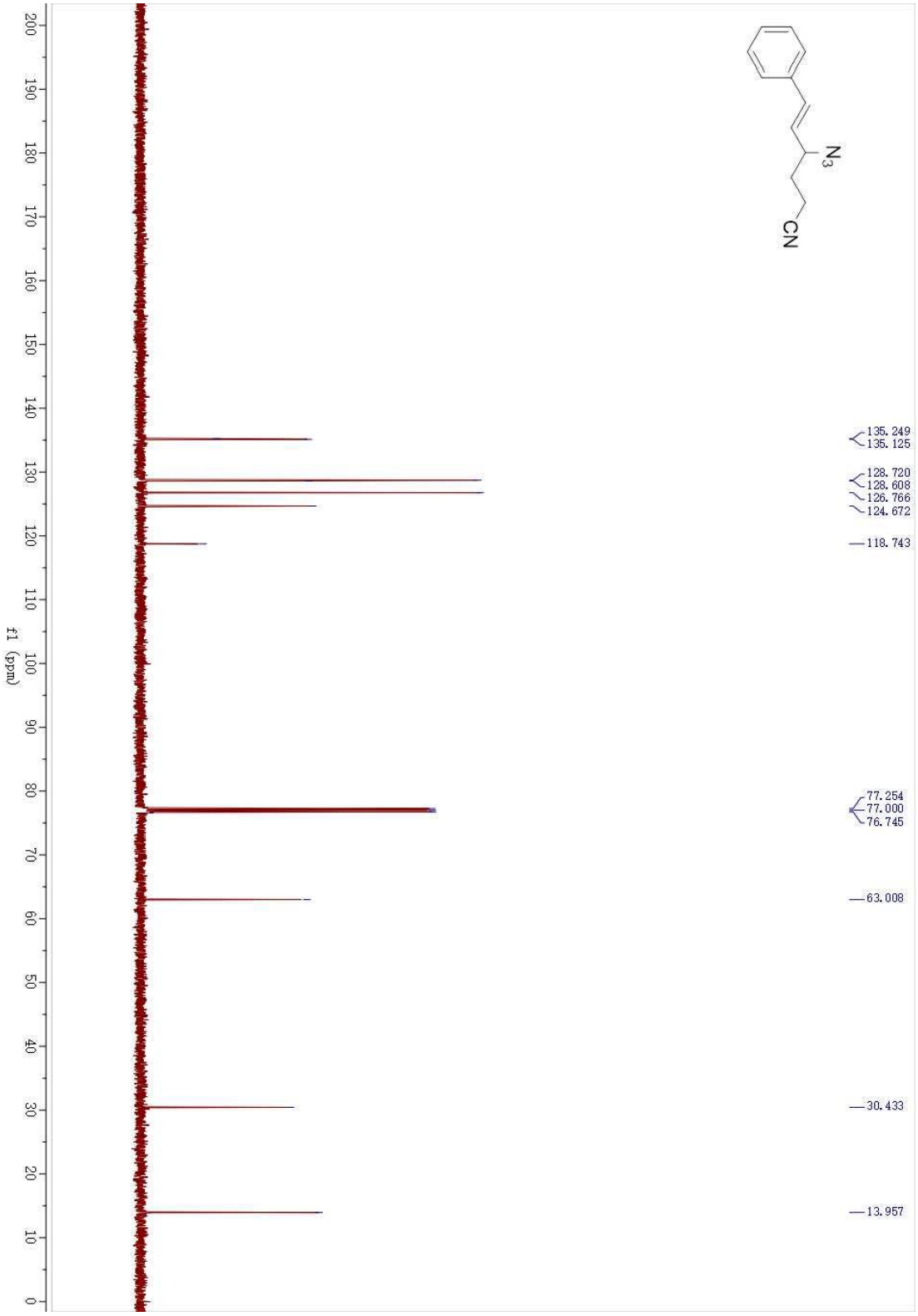
(*E*)-4-azido-6-(4-methoxyphenyl)hex-5-enenitrile (3a):



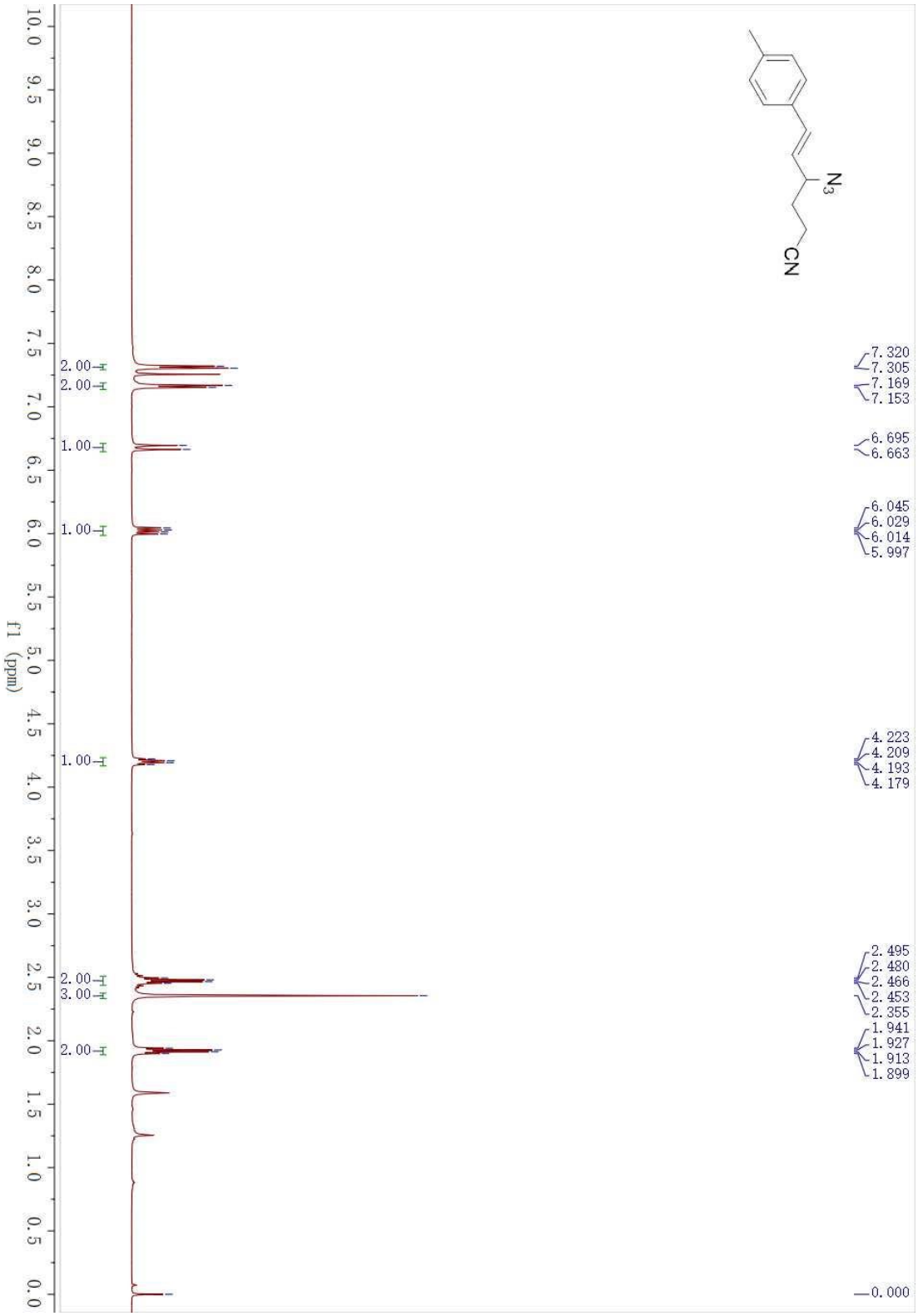

MeO
N3
CN
159.782
134.464
127.920
127.831
122.198
118.748
113.956
77.255
77.000
76.745
63.104
55.130
30.311
13.769
f1 (ppm)

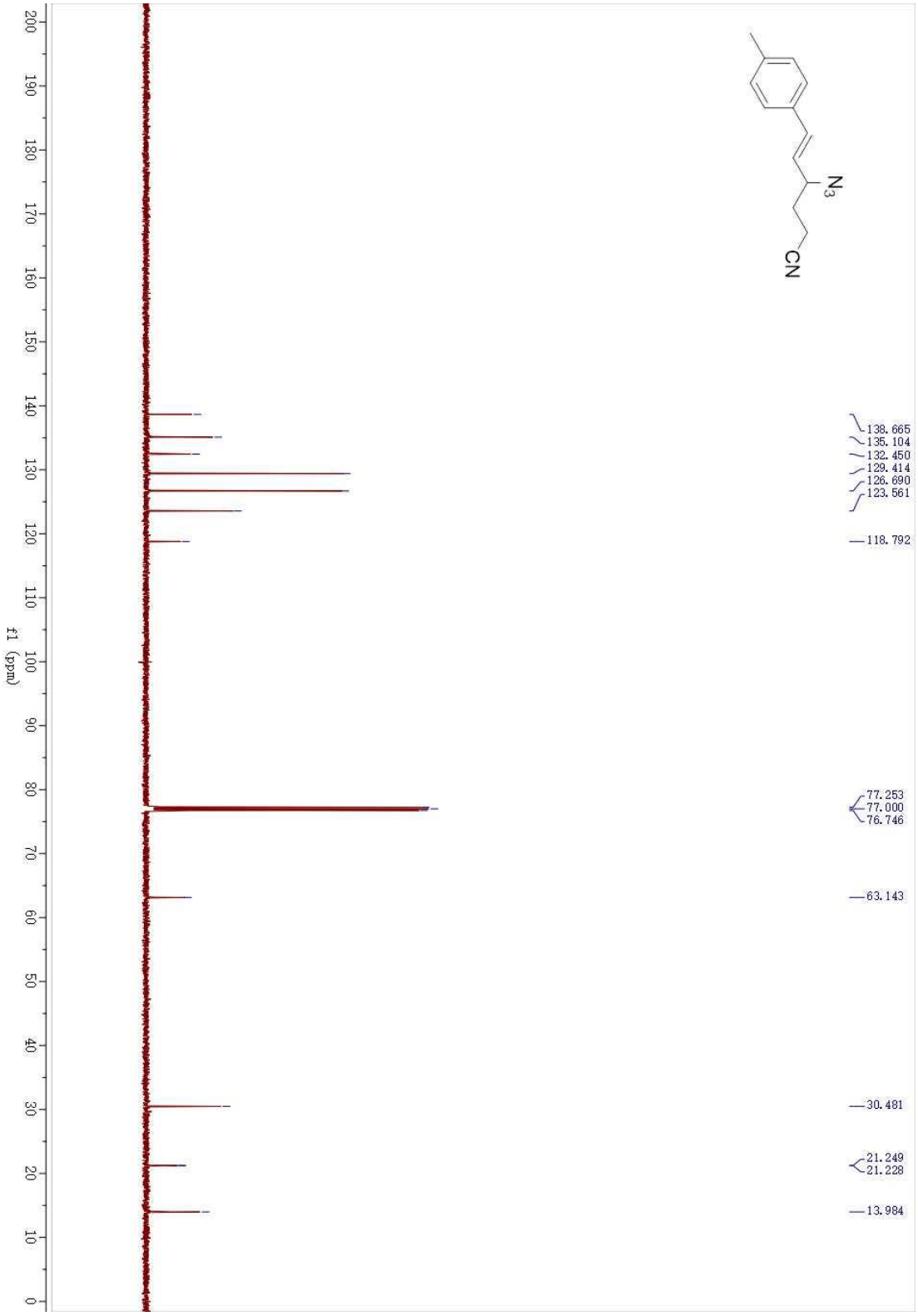
(*E*)-1-(hept-1-en-6-yn-1-yl)-4-methoxybenzene (3b):



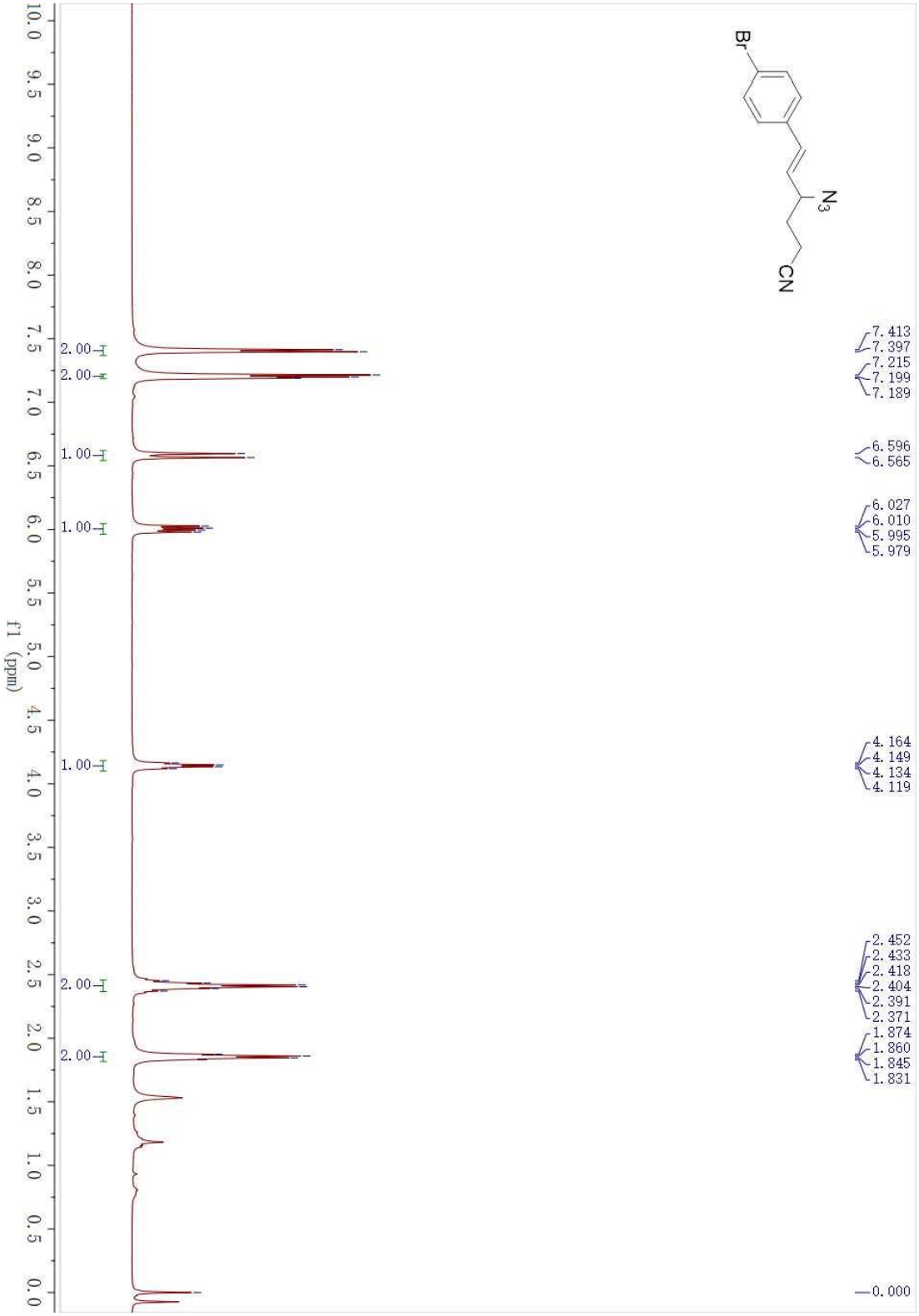

N3
CN
135.249
135.125
128.720
128.608
126.766
124.672
118.743
77.254
77.000
76.745
63.008
30.433
13.957
f1 (ppm)

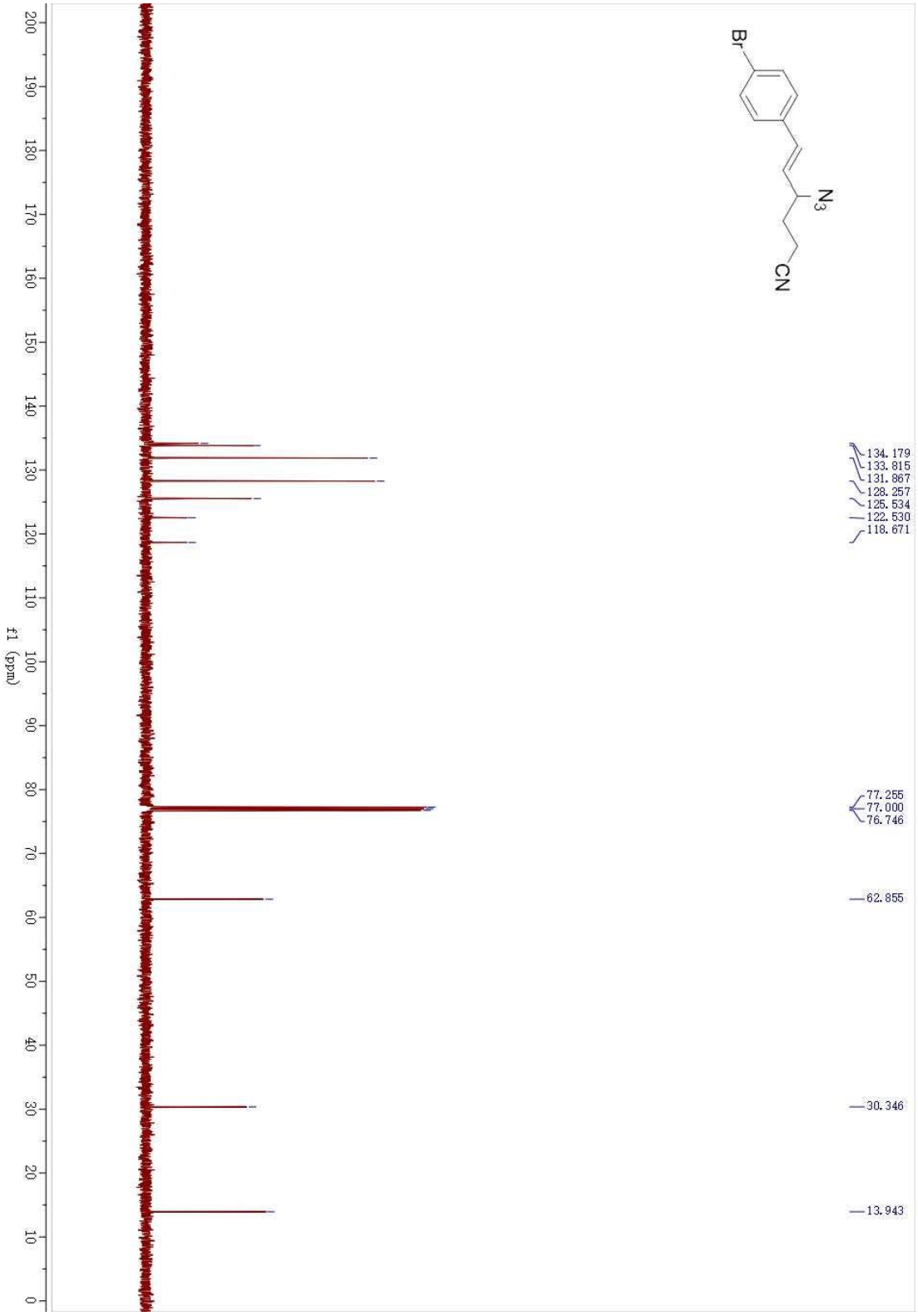
(*E*)-4-azido-6-(p-tolyl)hex-5-enenitrile (3c):



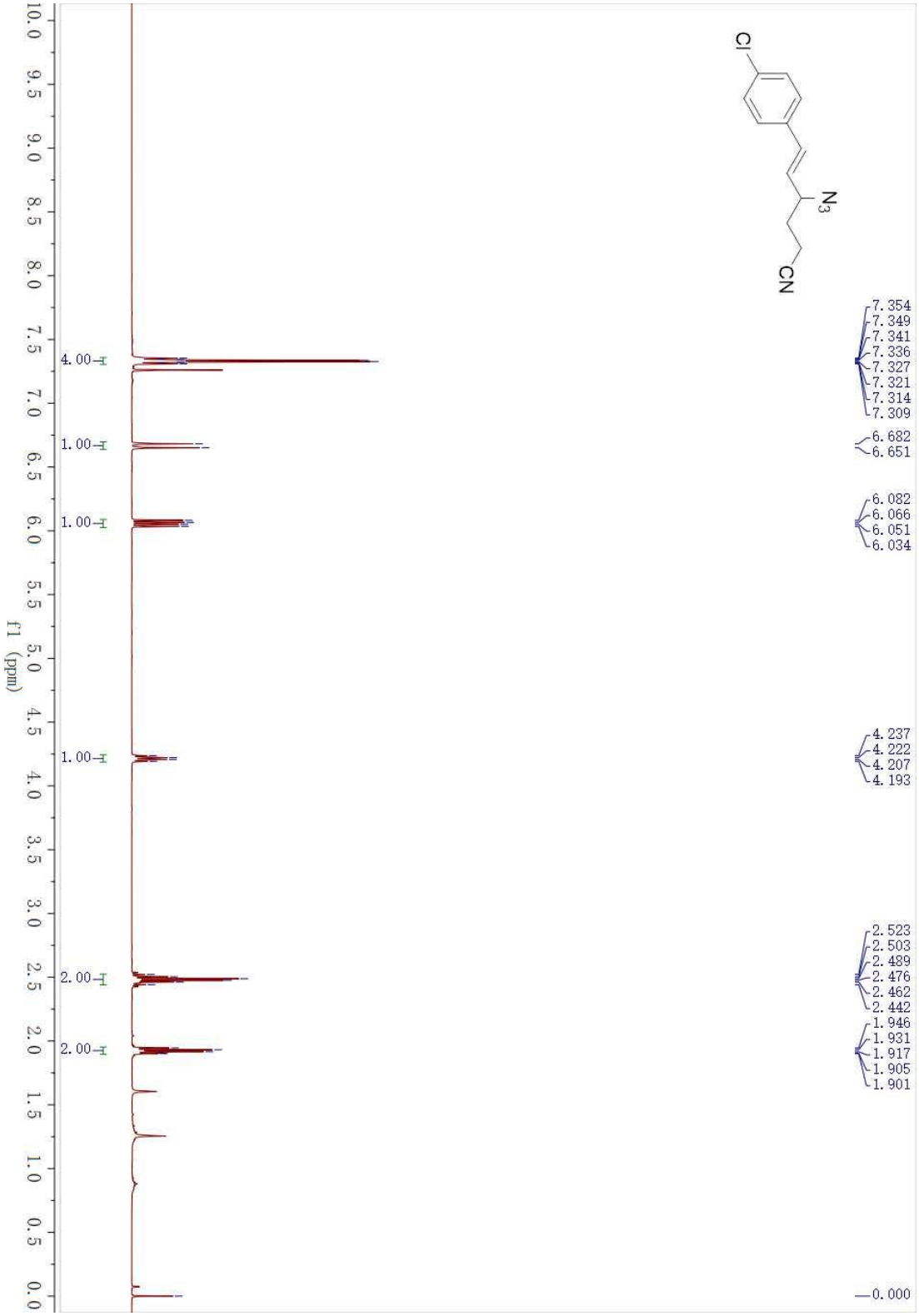

f1 (ppm)
138.665
135.104
132.450
129.414
126.690
123.561
118.792
77.253
77.000
76.746
63.143
30.481
21.249
21.228
13.984
N3
CN

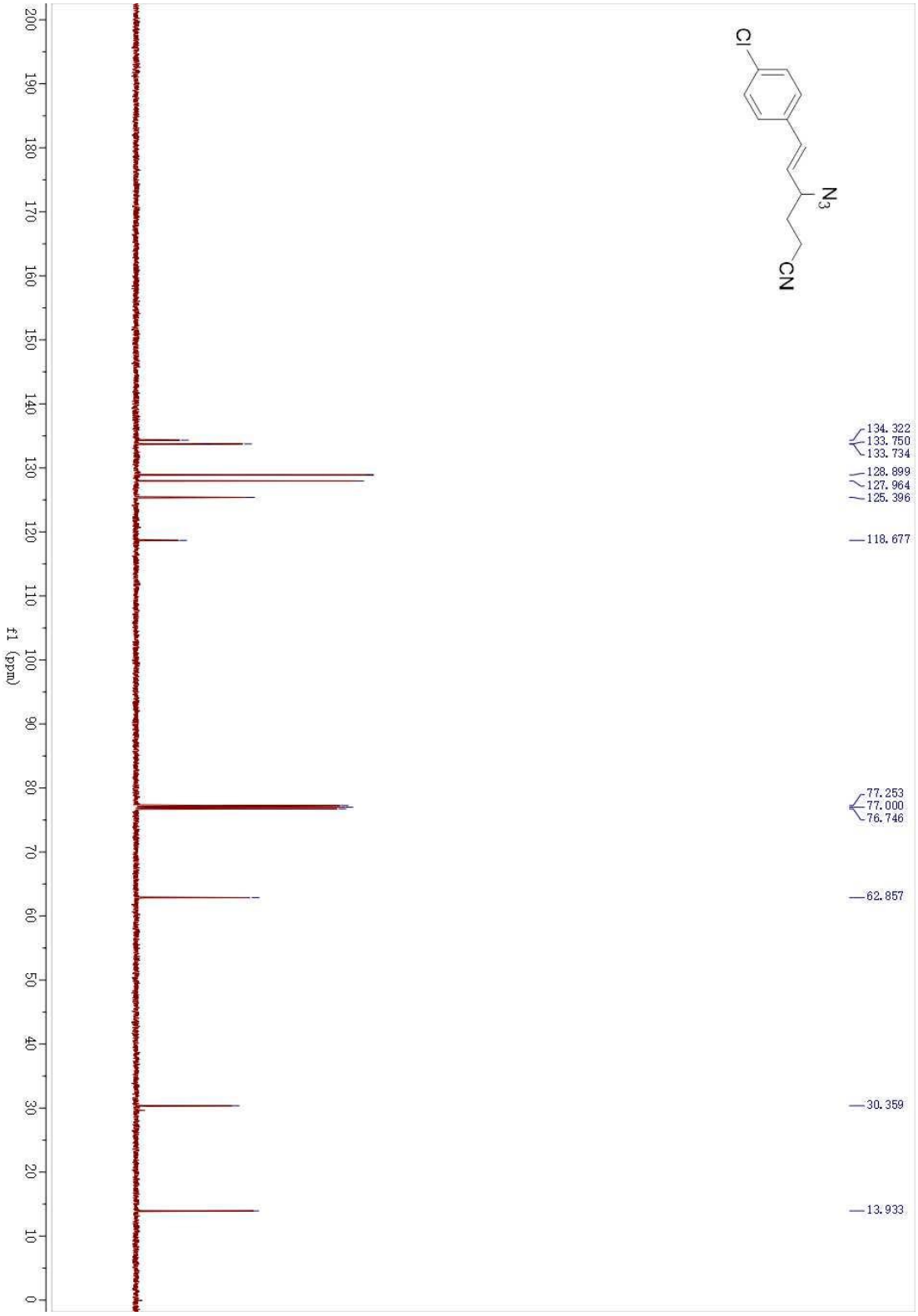
(*E*)-4-azido-6-(4-bromophenyl)hex-5-enenitrile (3d):



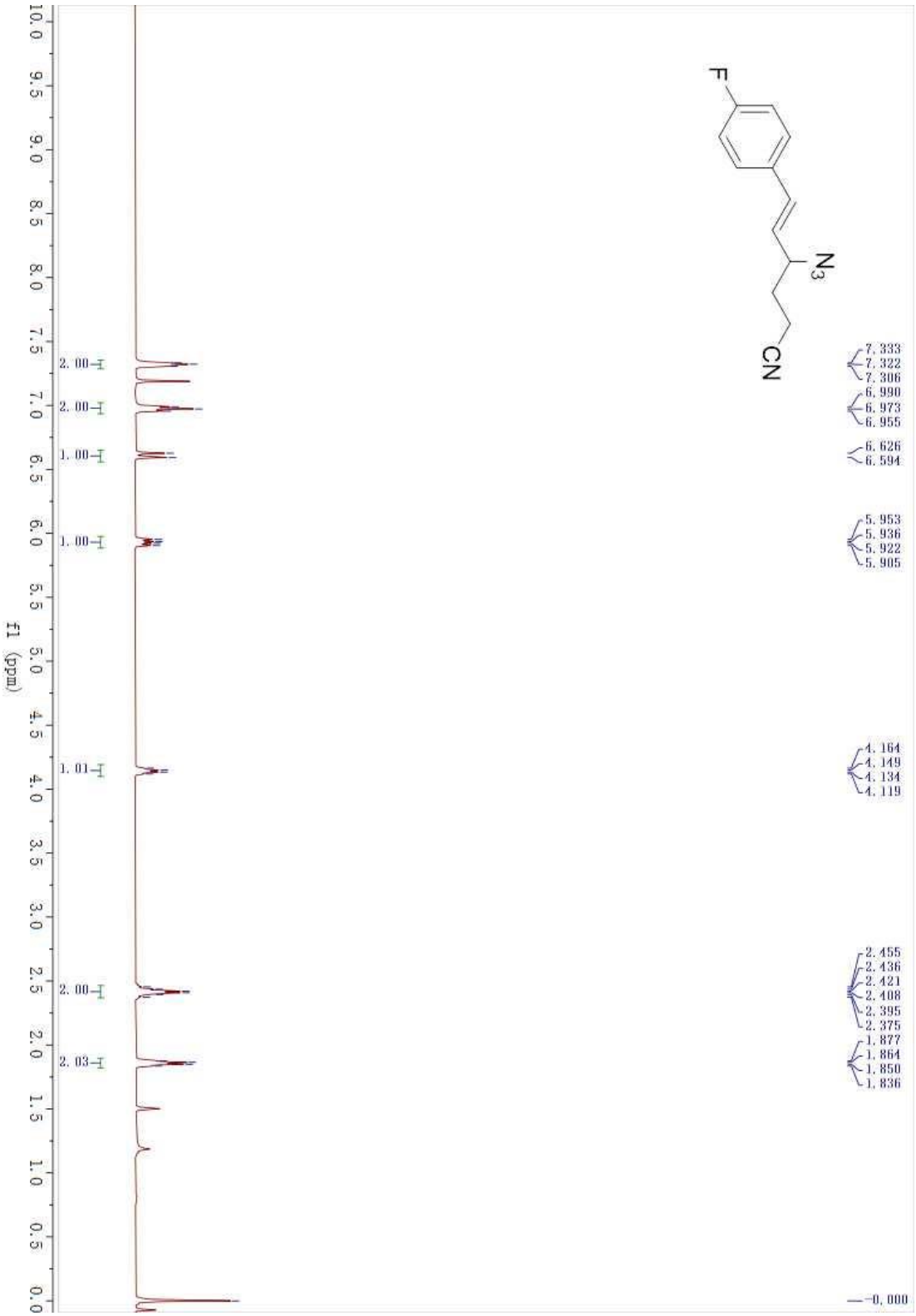

Br
N3
CN
134.179
133.815
131.867
128.257
125.534
122.530
118.671
77.255
77.000
76.746
62.855
30.346
13.943
f1 (ppm)

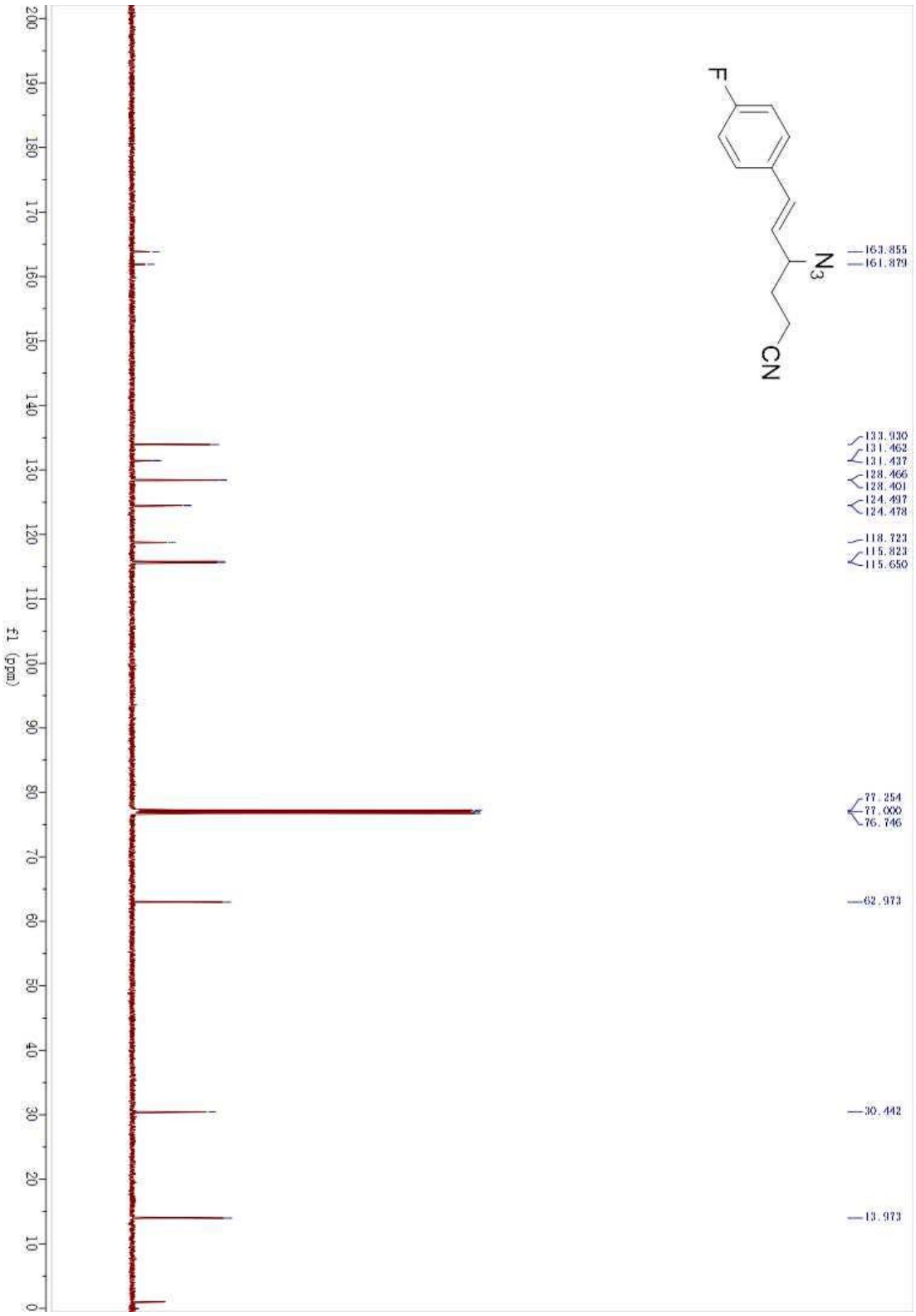
(*E*)-4-azido-6-(4-chlorophenyl)hex-5-enenitrile (3e):

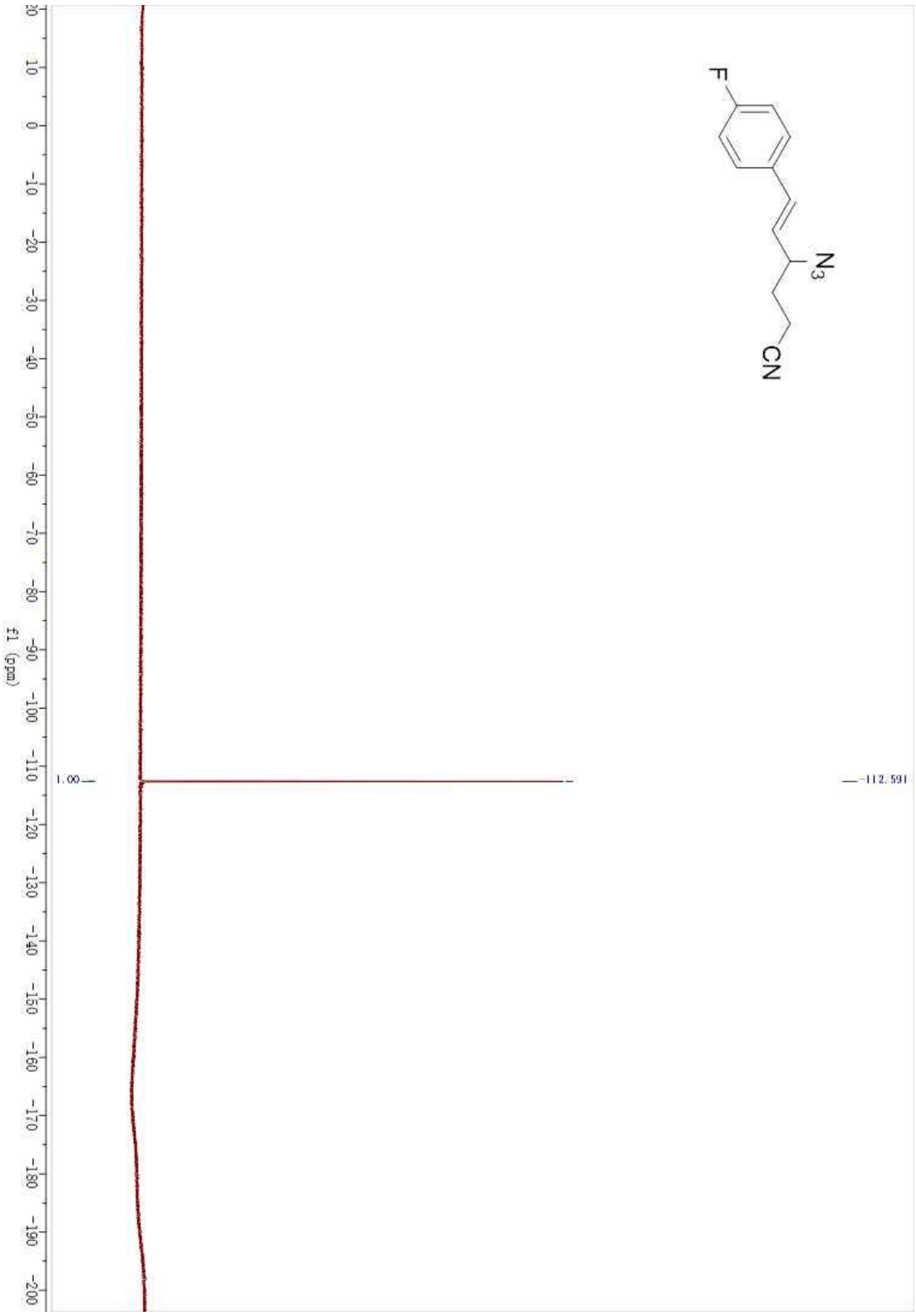



Cl
N3
CN
134.322
133.750
133.734
128.899
127.964
125.396
118.677
77.253
77.000
76.746
62.857
30.359
13.933
f1 (ppm)

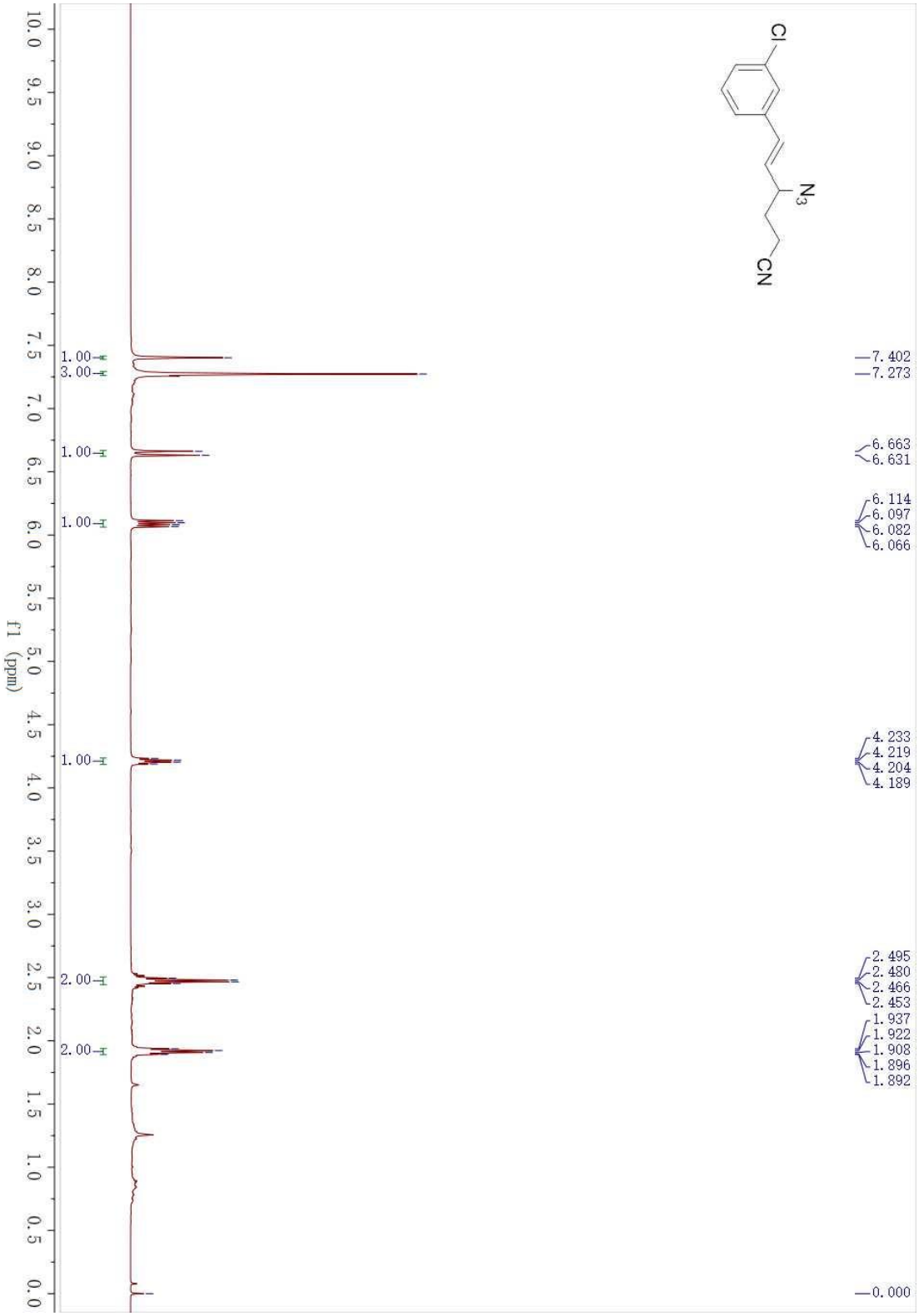
(*E*)-4-azido-6-(4-fluorophenyl)hex-5-enenitrile (3f):

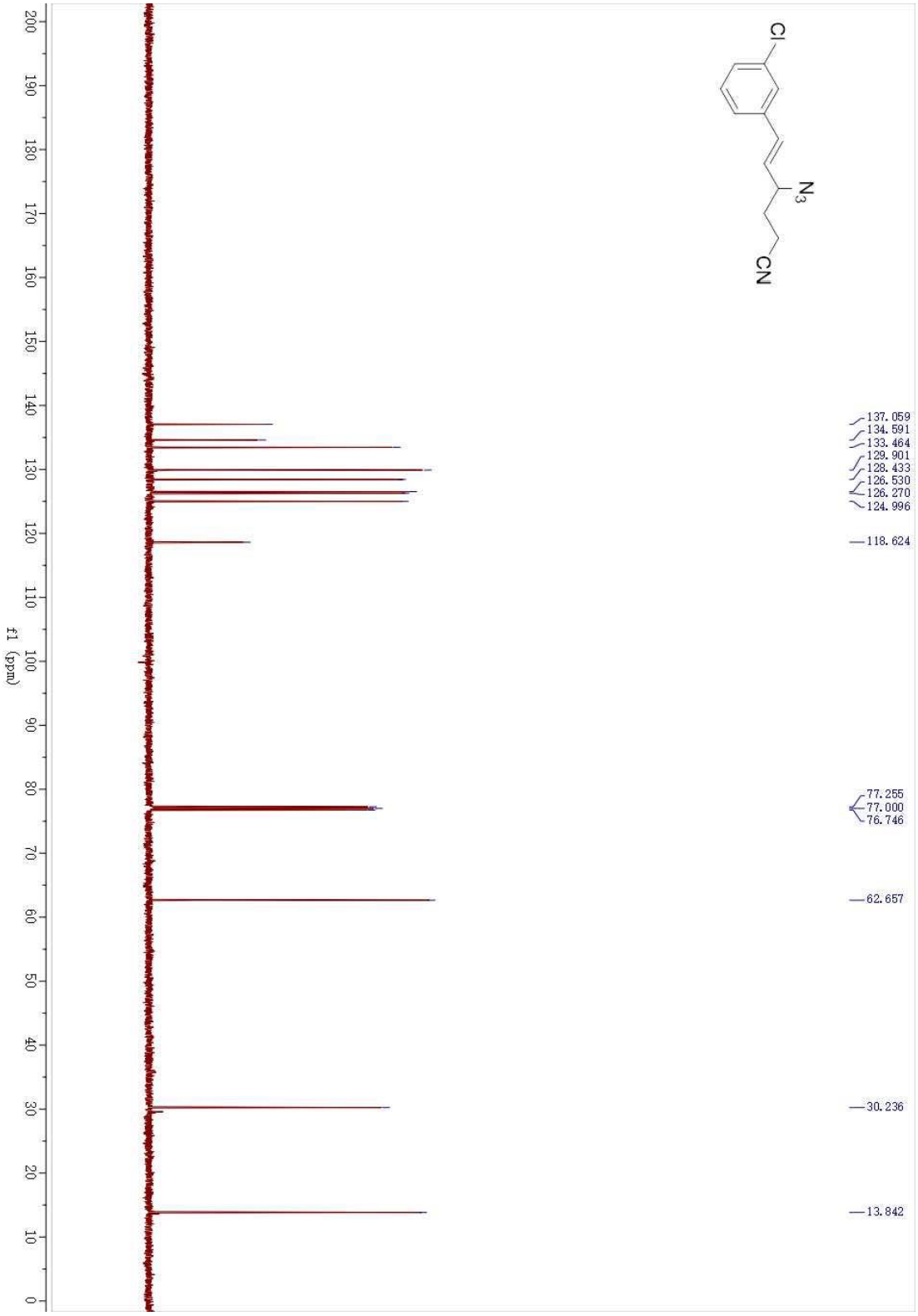



F
N3
CN
163.855
161.879
133.930
131.462
131.437
128.466
128.401
124.497
124.478
118.723
115.823
115.650
77.254
77.000
76.746
62.973
30.442
13.973
f1 (ppm)

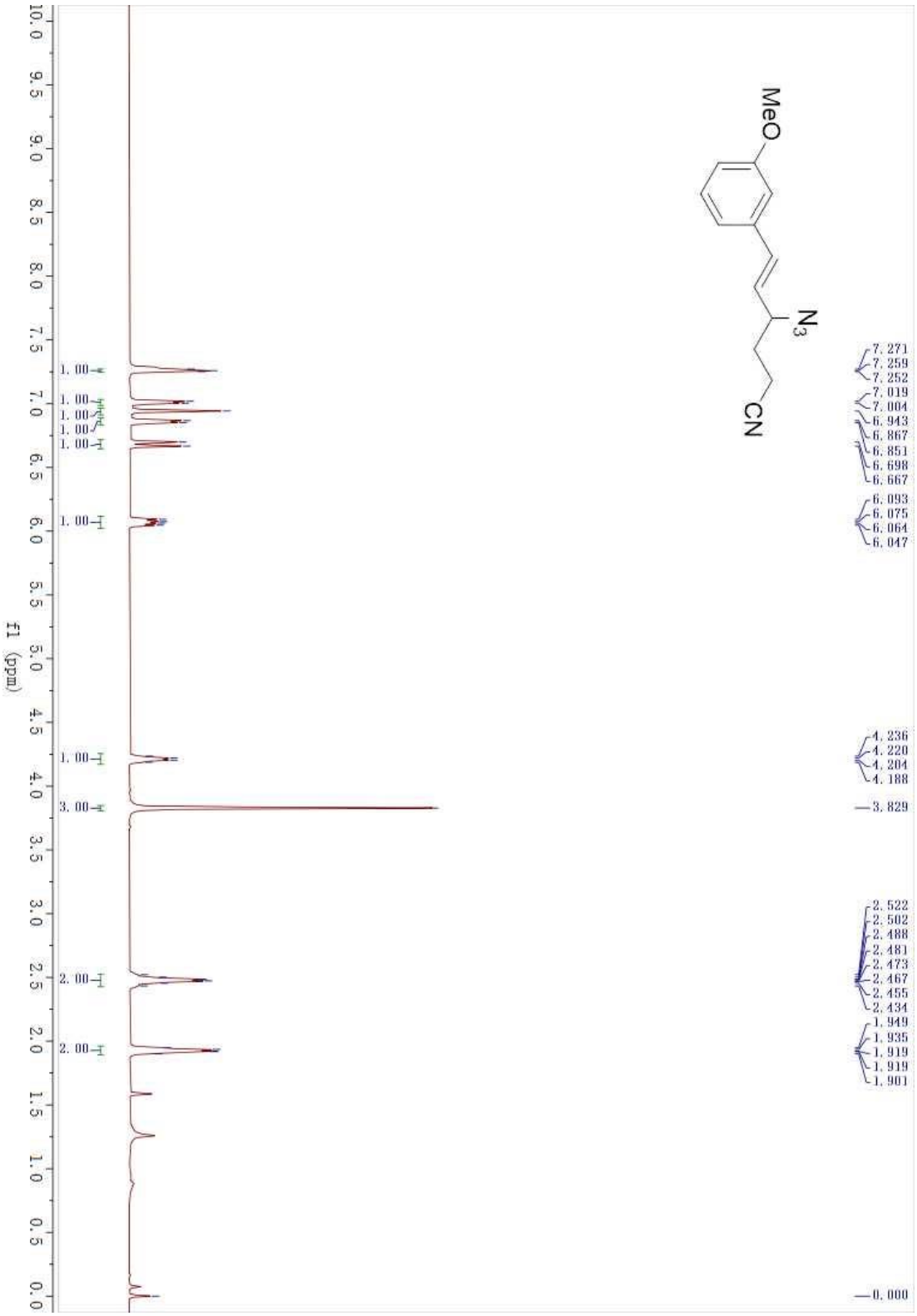

F
N3
CN
f1 (ppm)
1.00
-112.591

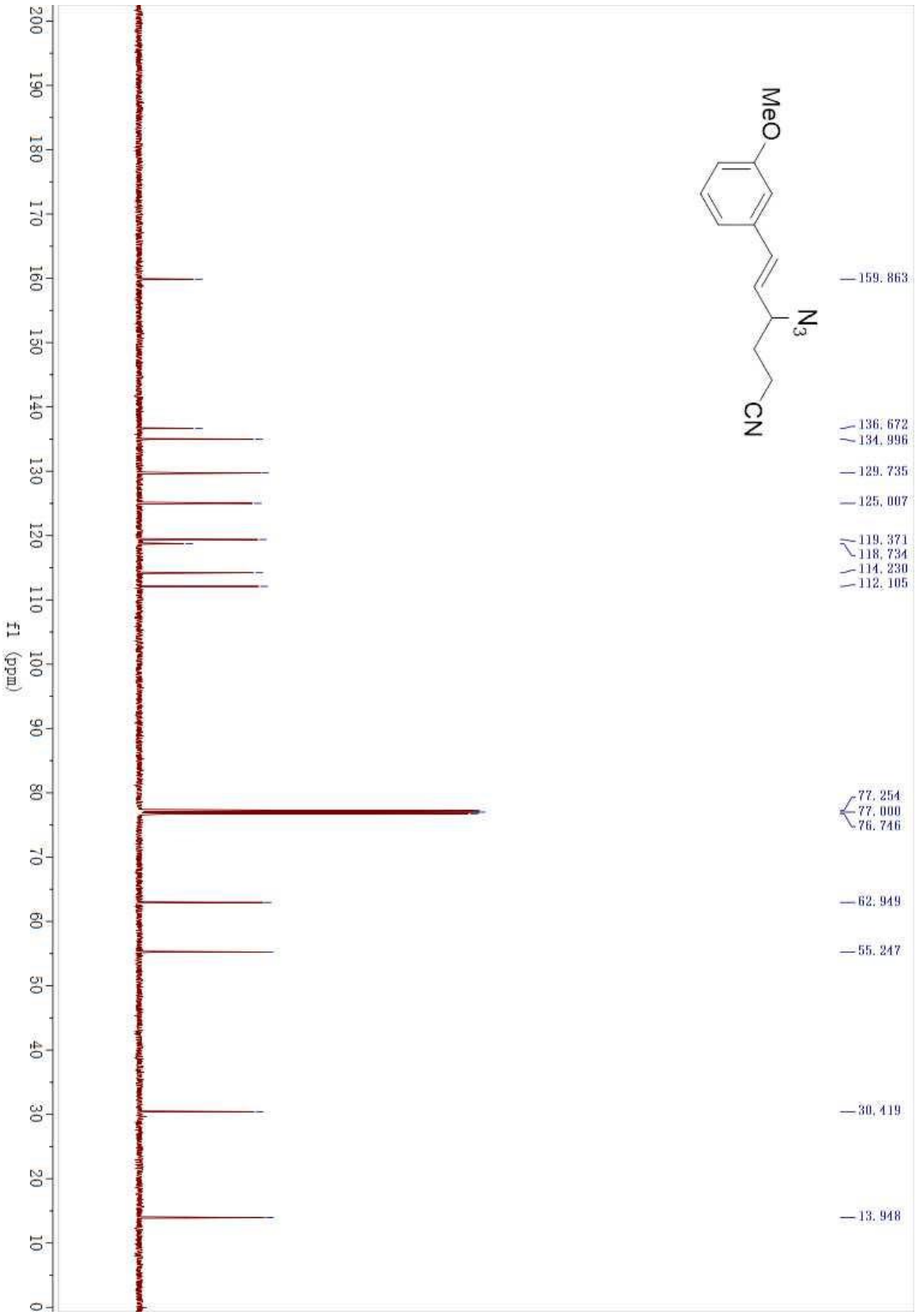
(*E*)-4-azido-6-(3-chlorophenyl)hex-5-enenitrile (3g):



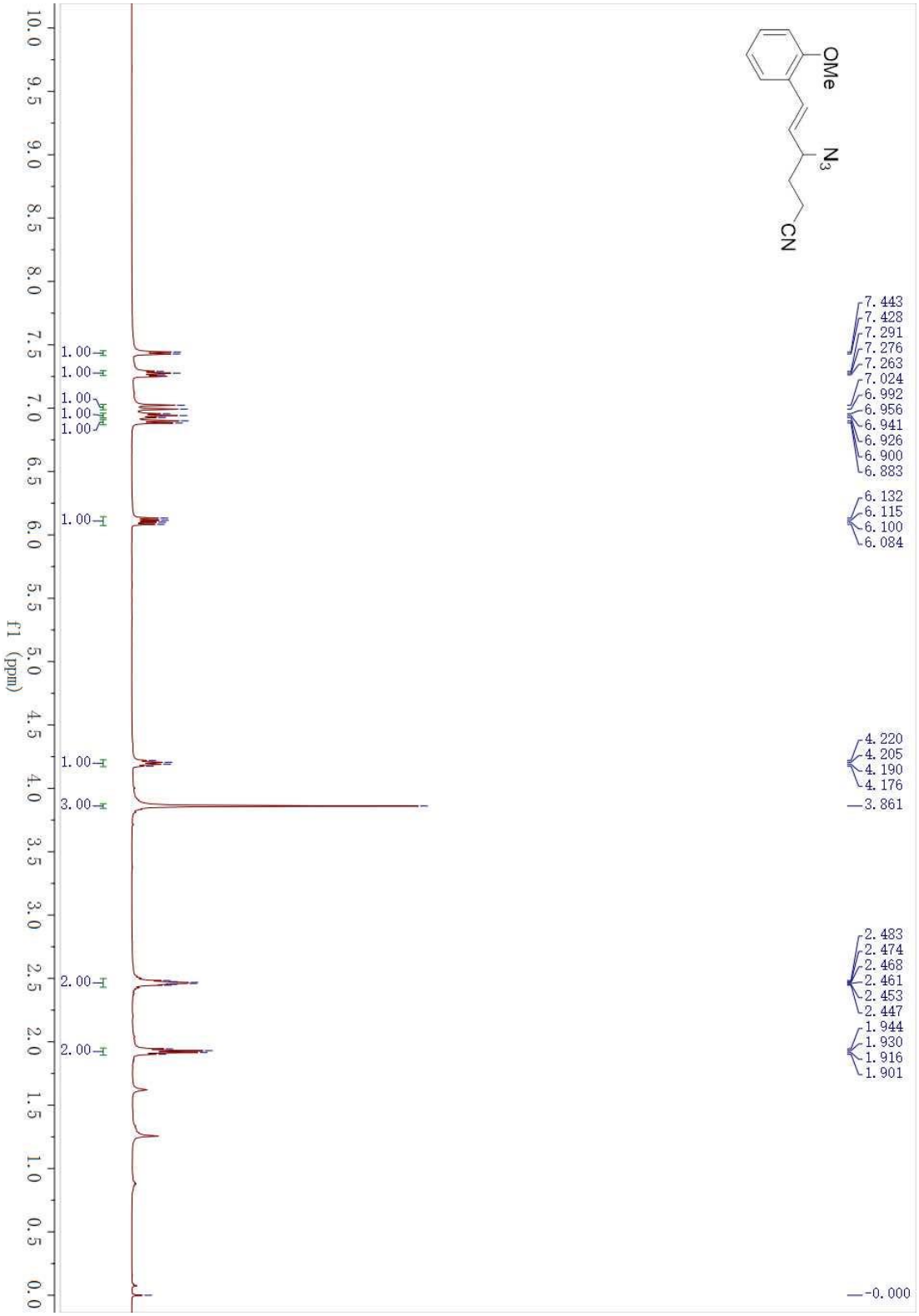

Cl
N3
CN
137.059
134.591
133.464
129.901
128.433
126.530
126.270
124.996
118.624
77.255
77.000
76.746
62.657
30.236
13.842
f1 (ppm)

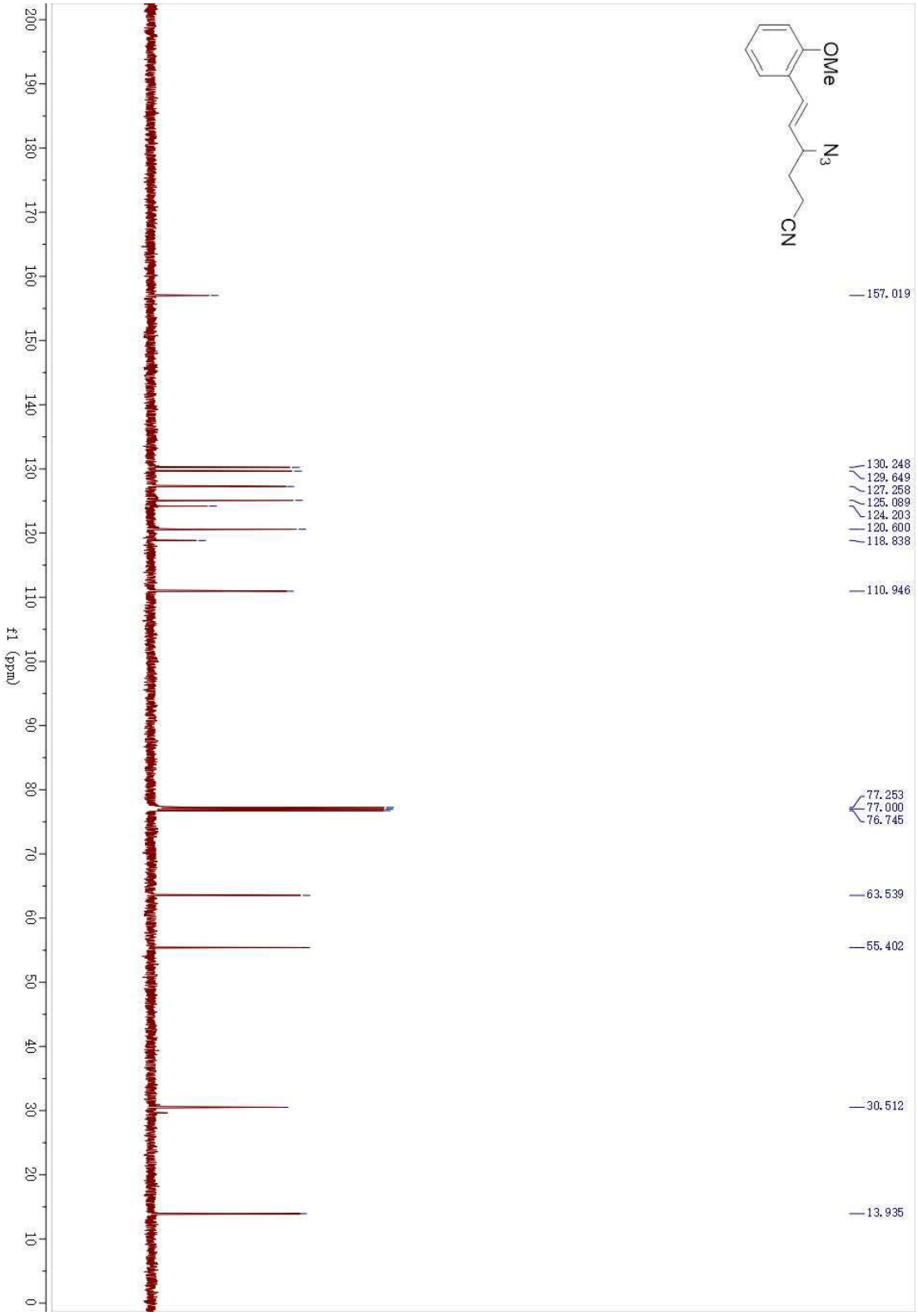
(*E*)-4-azido-6-(3-methoxyphenyl)hex-5-enenitrile (3h):



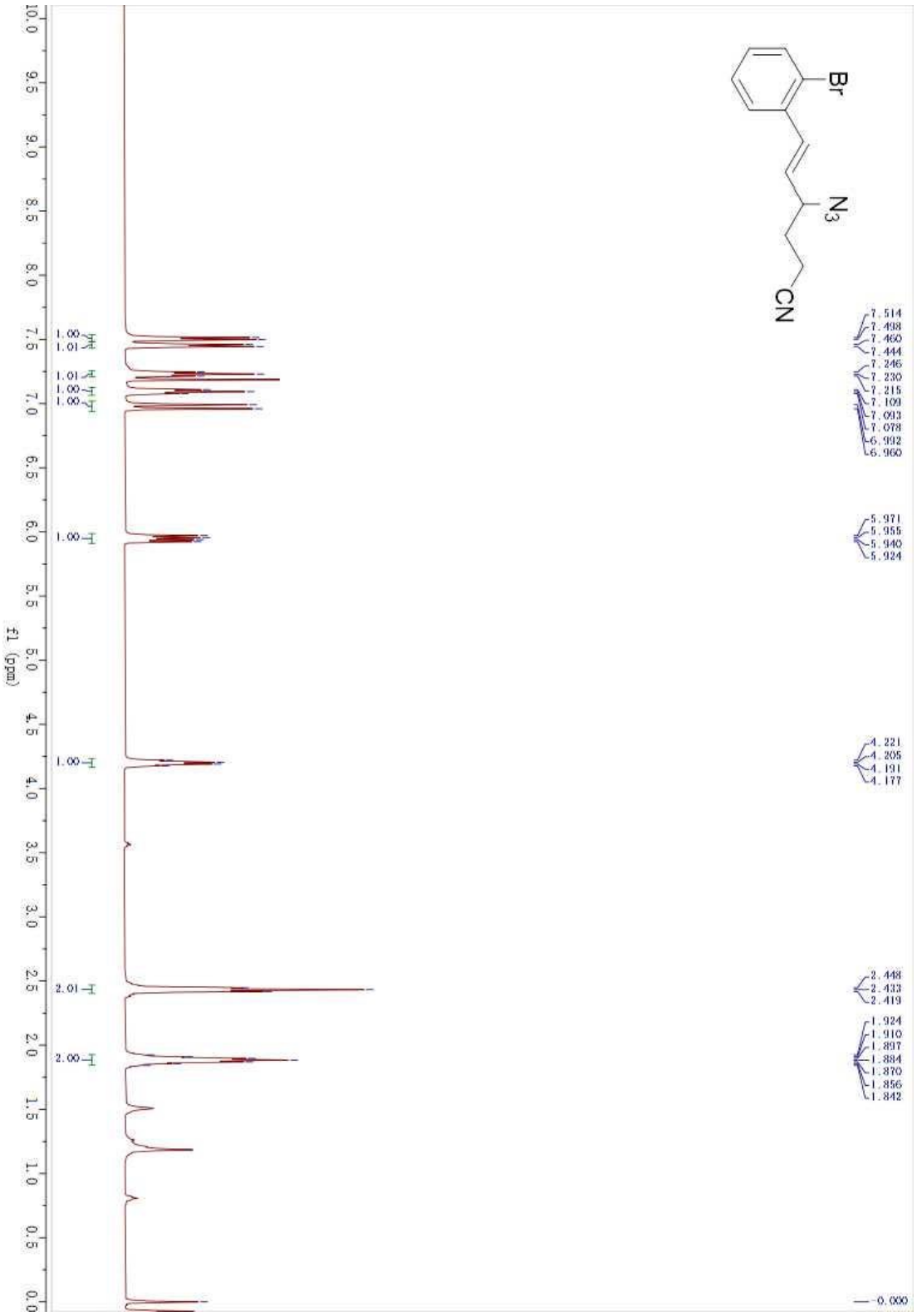

MeO
N3
CN
159.863
136.672
134.996
129.735
125.007
119.371
118.734
114.230
112.105
77.254
77.000
76.746
62.949
55.247
30.419
13.948
f1 (ppm)

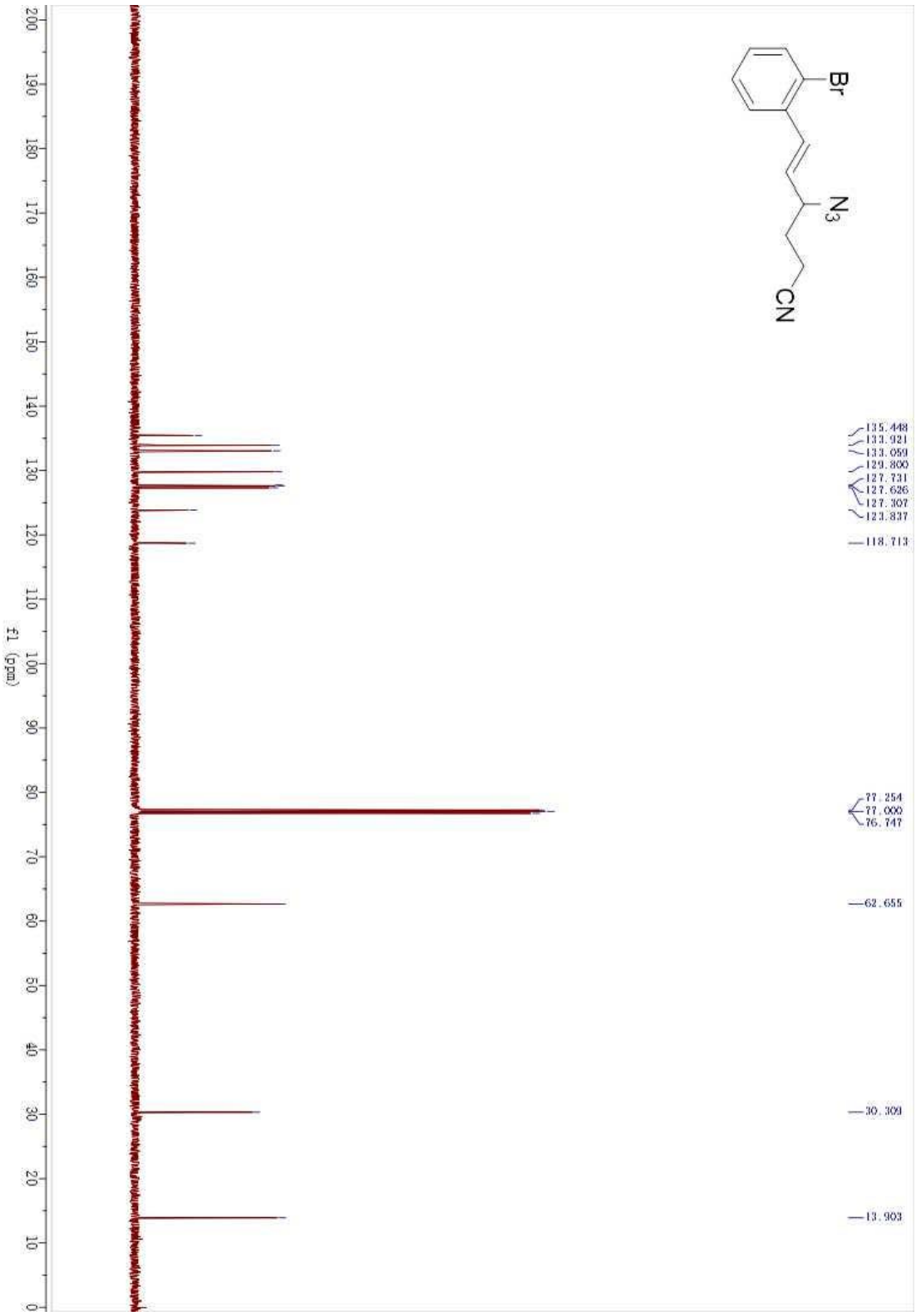
(*E*)-4-azido-6-(2-methoxyphenyl)hex-5-enenitrile (3i):



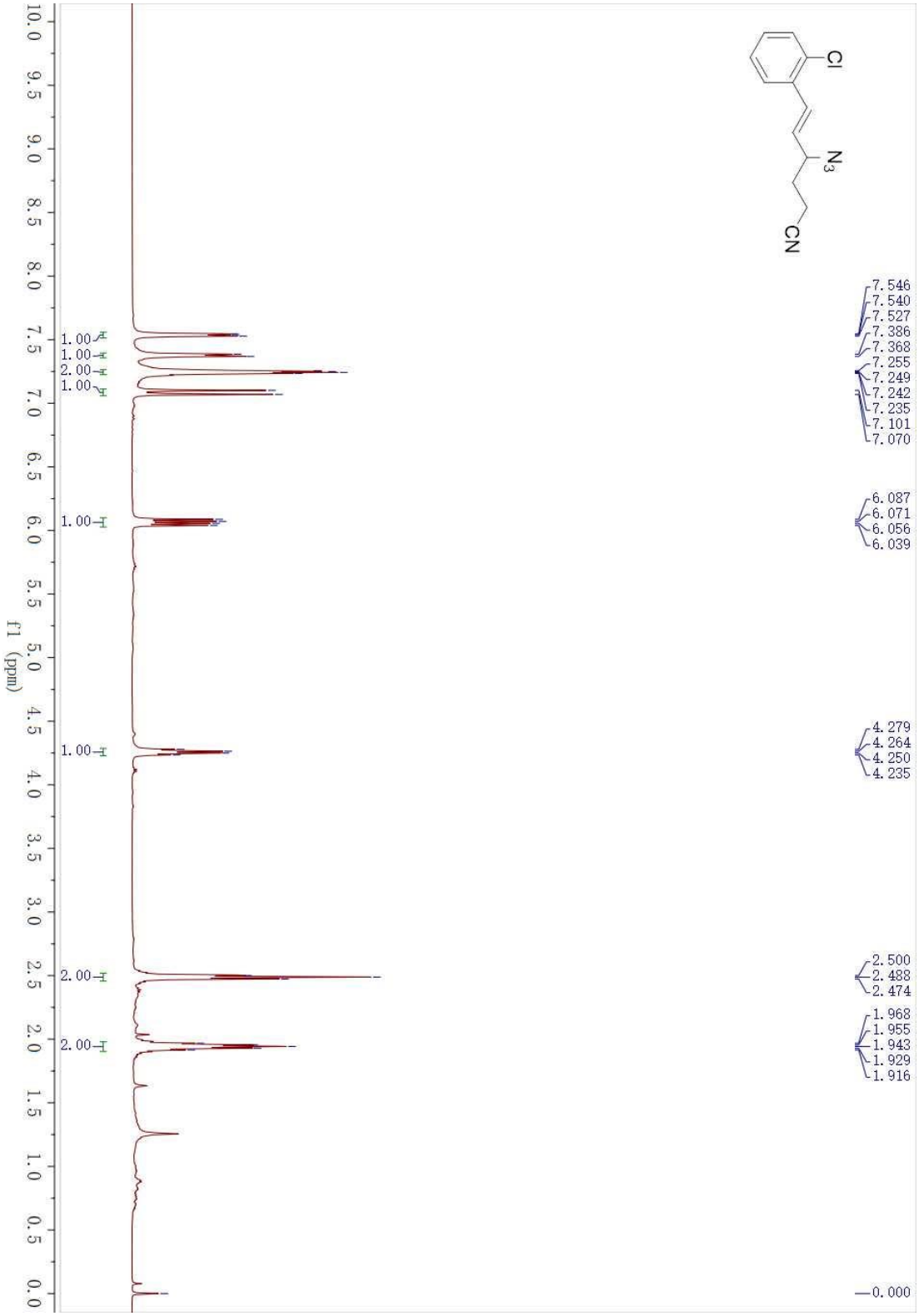

OMe
N3
CN
157.019
130.248
129.649
127.258
125.089
124.203
120.600
118.838
110.946
77.253
77.000
76.745
63.539
55.402
30.512
13.935
f1 (ppm)

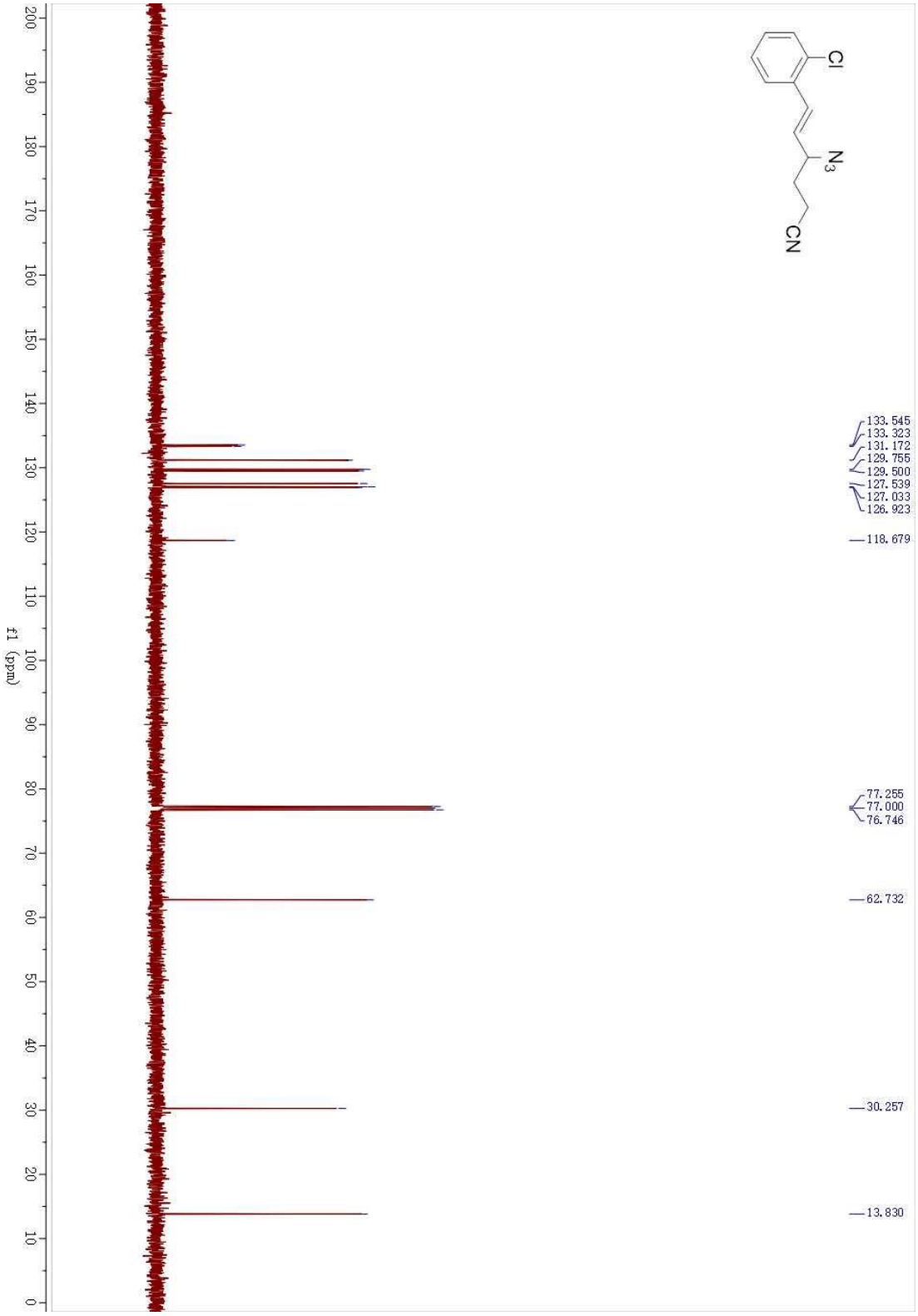
(*E*)-4-azido-6-(2-bromophenyl)hex-5-enenitrile (3j):



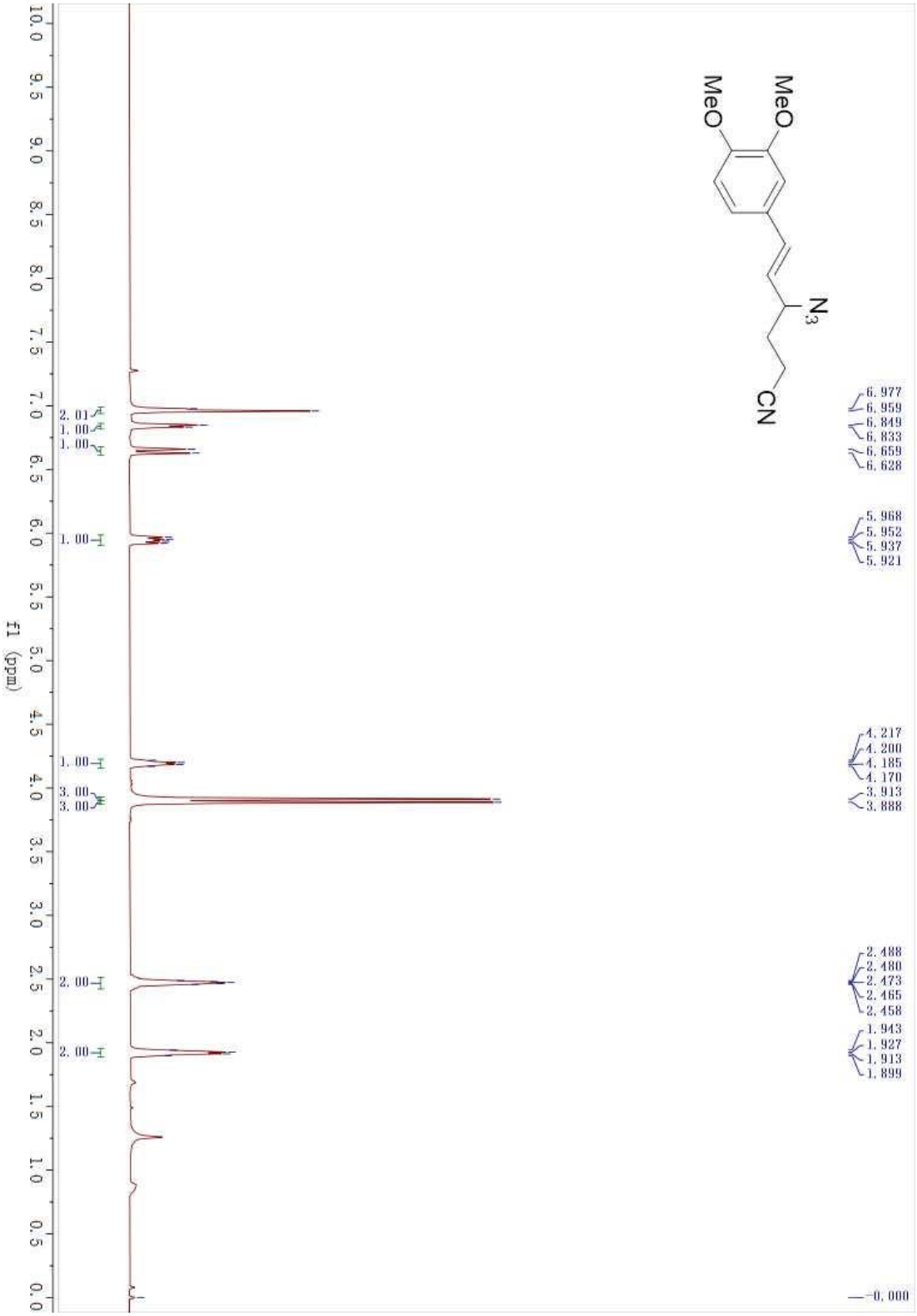

Br
N3
CN
135.448
133.921
133.059
129.800
127.731
127.626
127.307
123.837
118.713
77.254
77.000
76.747
62.655
30.309
13.903
f1 (ppm)

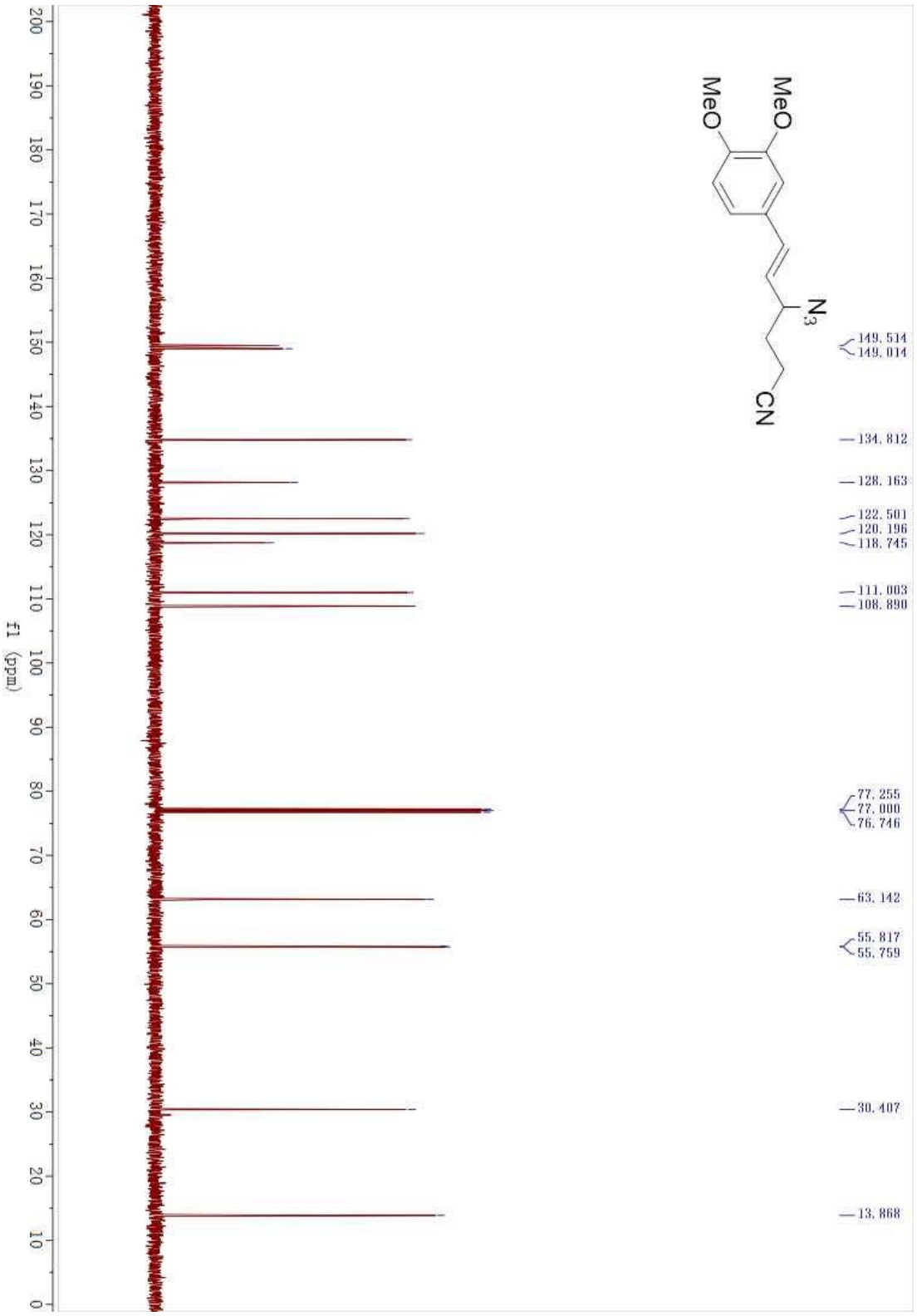
(*E*)-4-azido-6-(2-chlorophenyl)hex-5-enenitrile (3k):



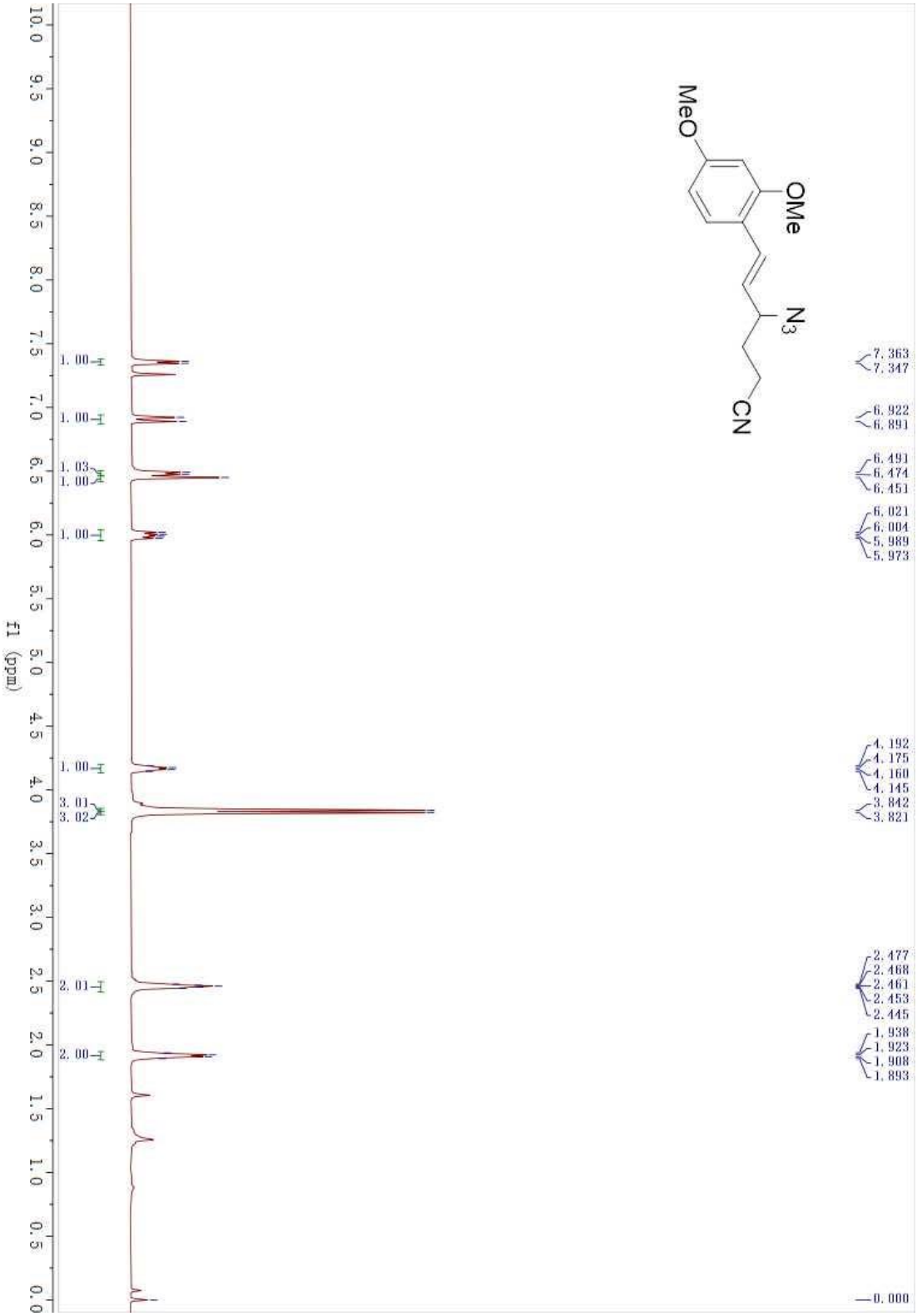

Cl
N3
CN
133.545
133.323
131.172
129.755
129.500
127.539
127.033
126.923
118.679
77.255
77.000
76.746
62.732
30.257
13.830
f1 (ppm)

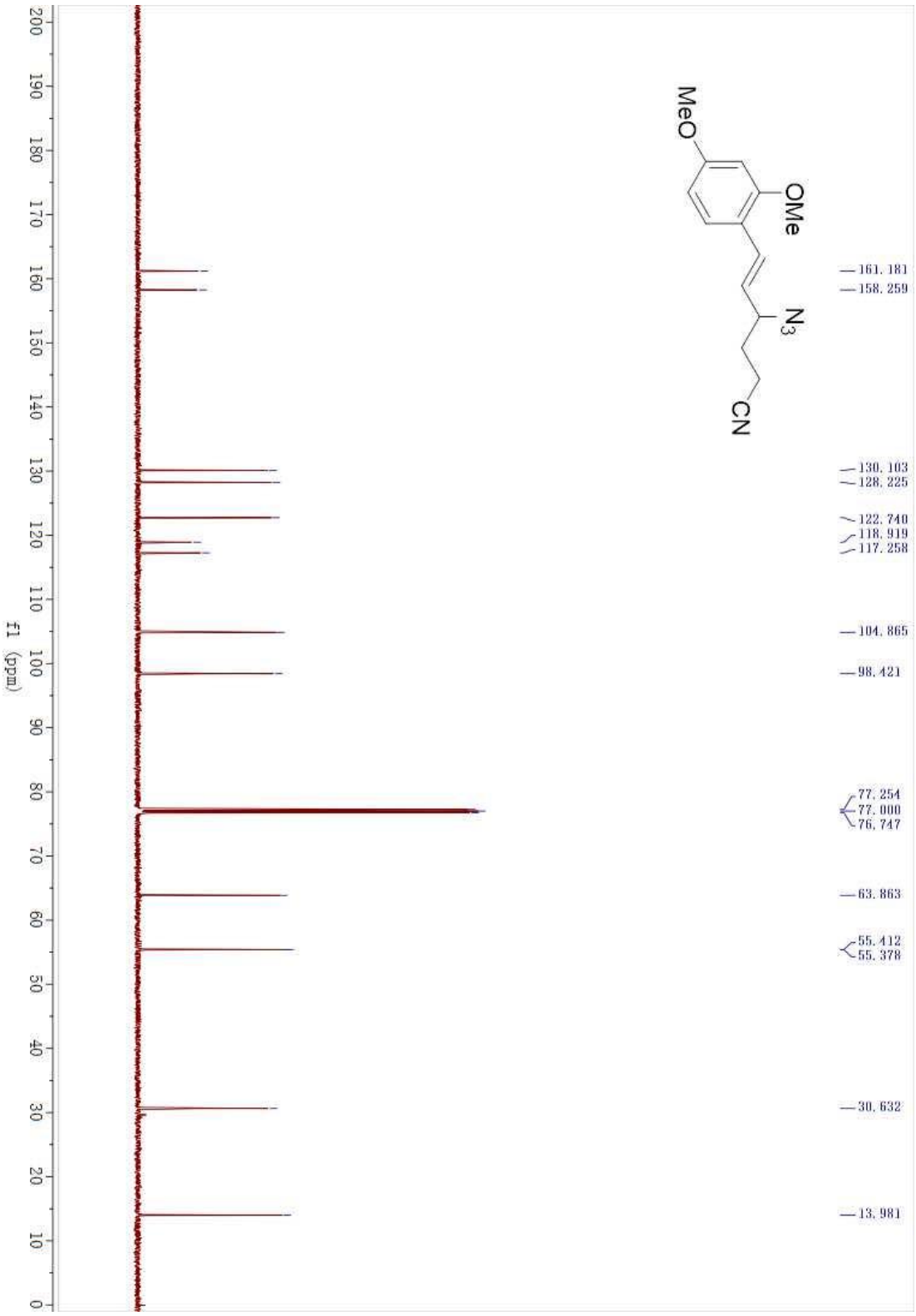
(*E*)-4-azido-6-(3,4-dimethoxyphenyl)hex-5-enenitrile (3l):



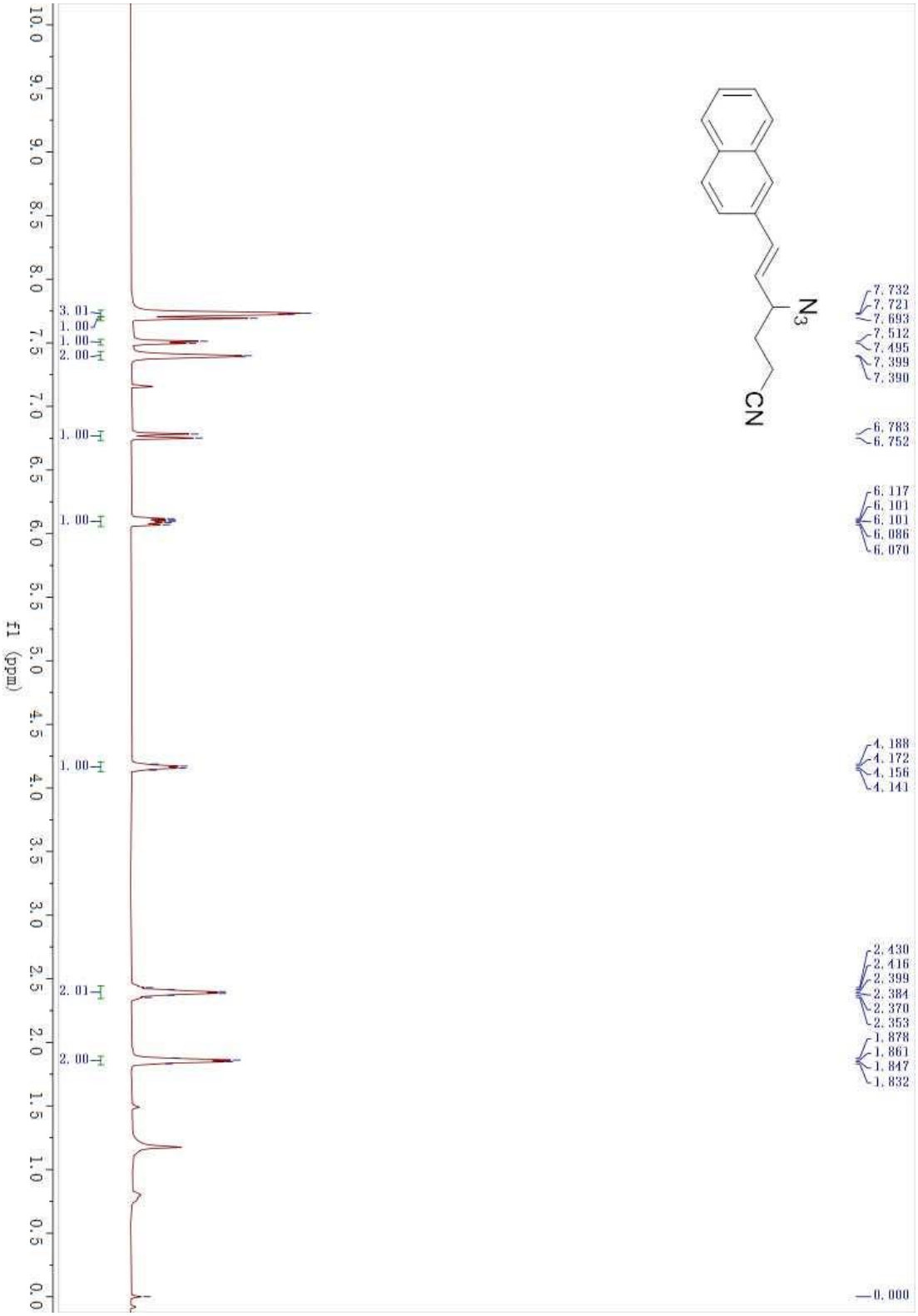

MeO
MeO
N3
CN
149.514
149.014
134.812
128.163
122.501
120.196
118.745
111.003
108.890
77.255
77.000
76.746
63.142
55.817
55.759
30.407
13.868
f1 (ppm)

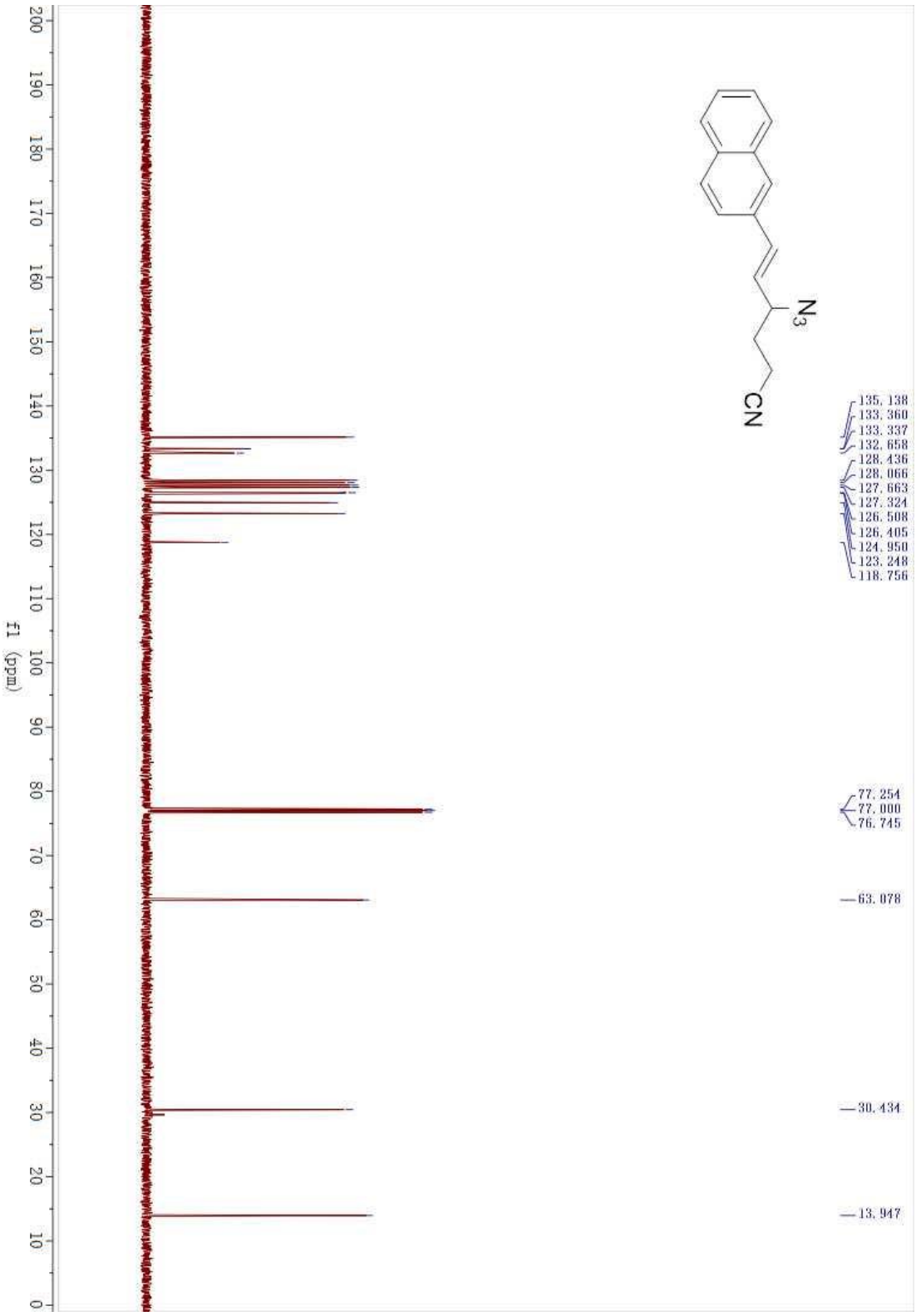
(*E*)-4-azido-6-(2,4-dimethoxyphenyl)hex-5-enenitrile (3m):



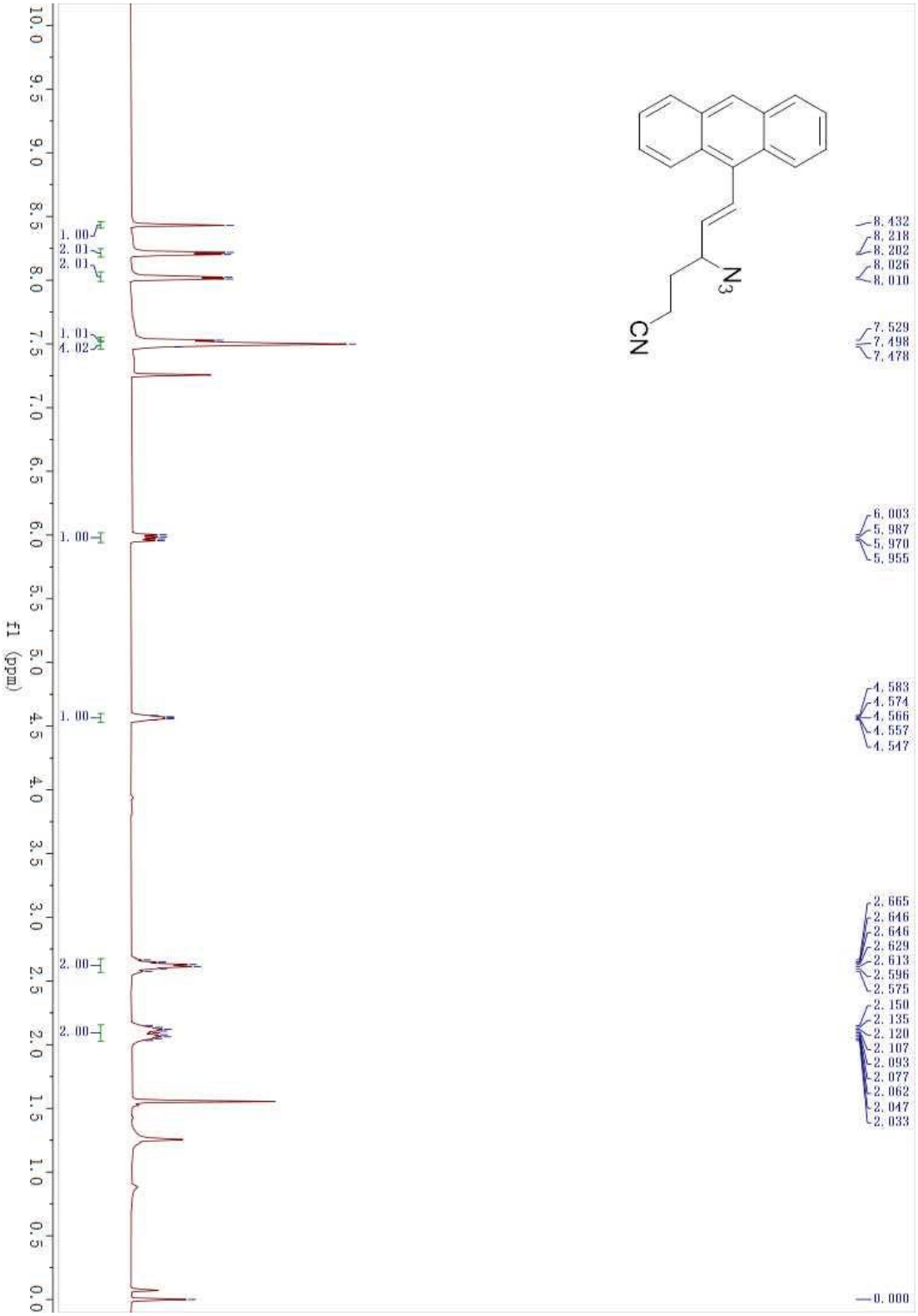

MeO
OMe
N3
CN
161.181
158.259
130.103
128.225
122.740
118.919
117.258
104.865
98.421
77.254
77.000
76.747
63.863
55.412
55.378
30.632
13.981
f1 (ppm)

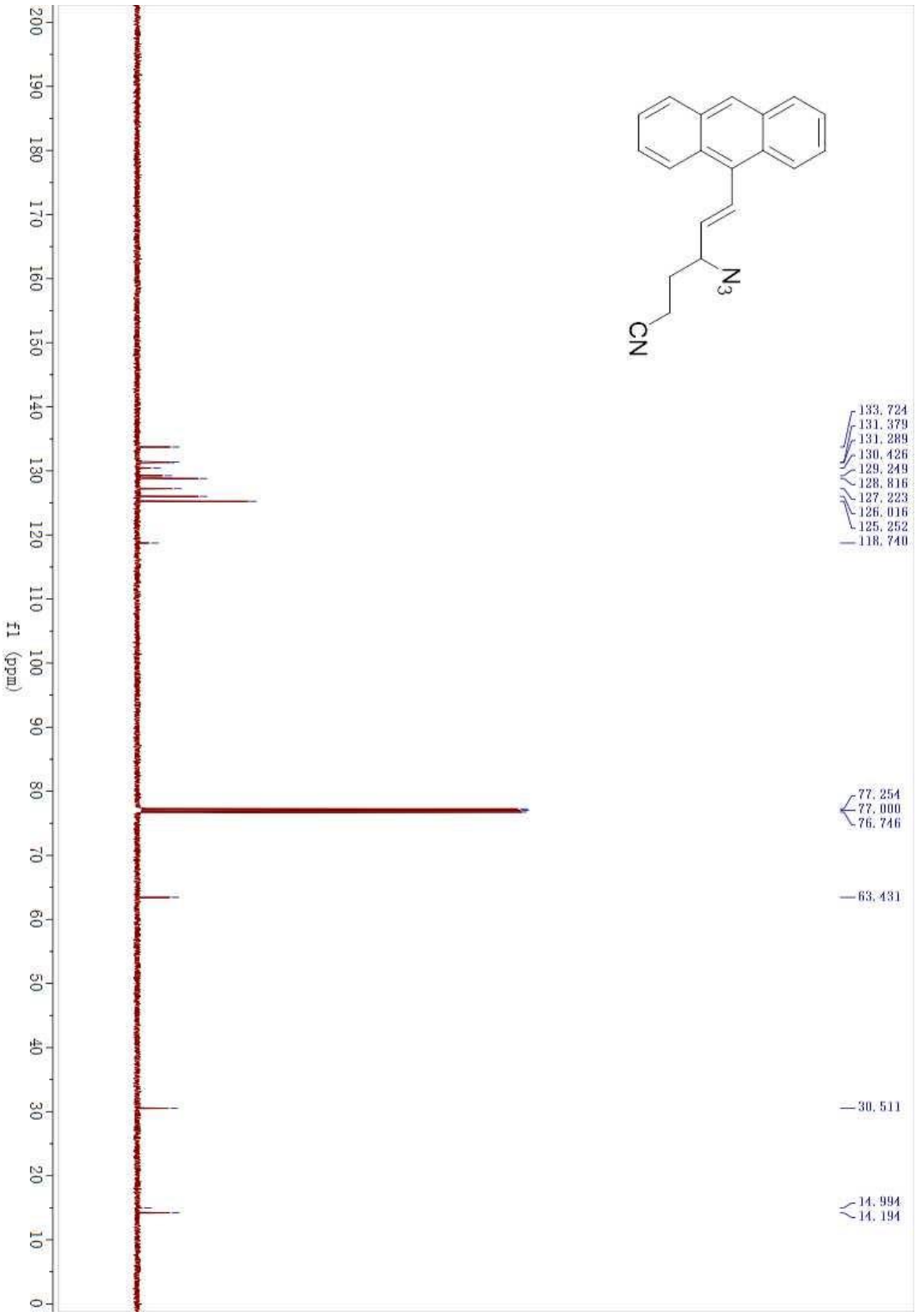
(*E*)-4-azido-6-(naphthalen-2-yl)hex-5-enenitrile (3n):



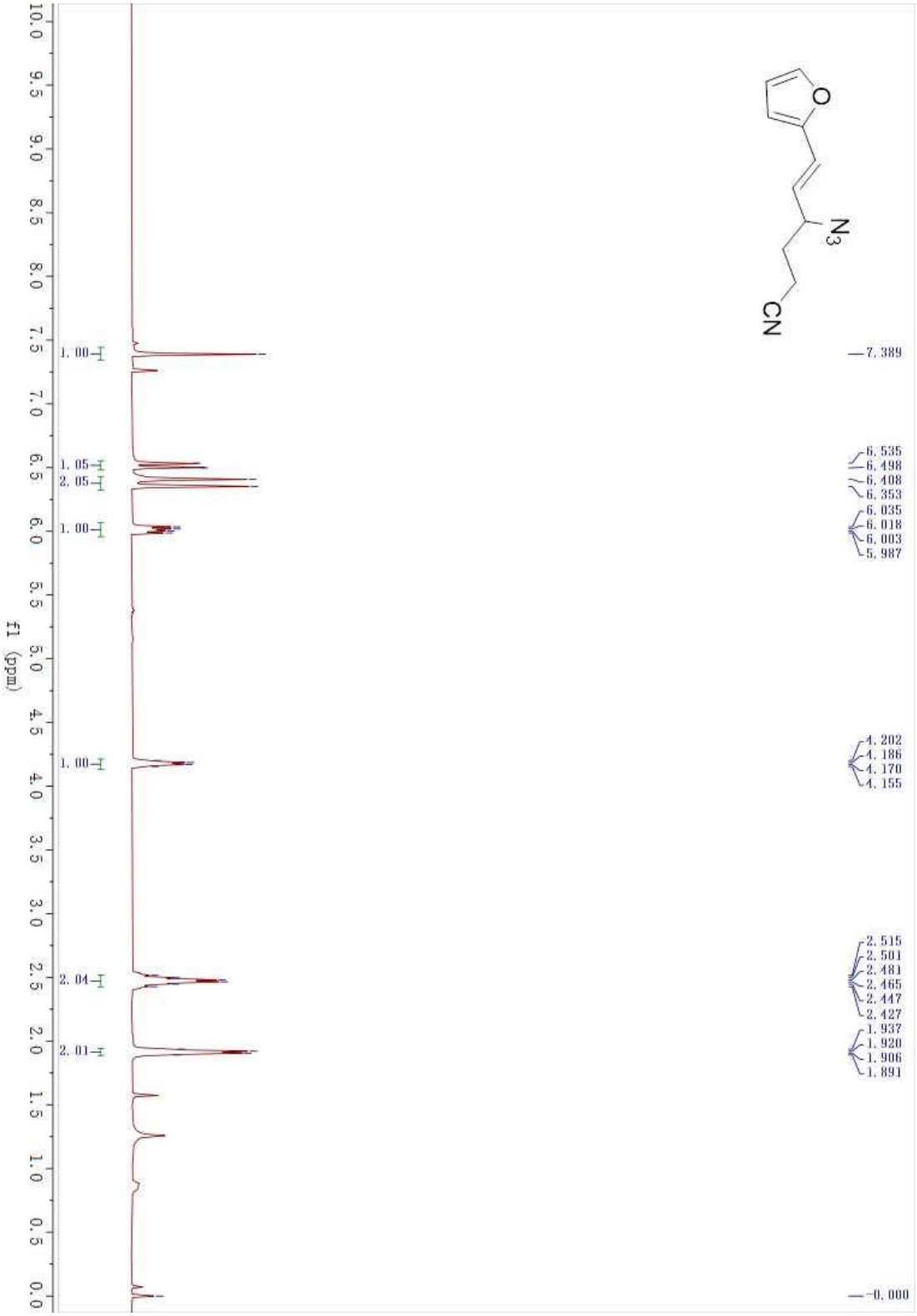

135.138
133.360
133.337
132.658
128.436
128.066
127.663
127.324
126.508
126.405
124.950
123.248
118.756
77.254
77.000
76.745
63.078
30.434
13.947
N3
CN
f1 (ppm)

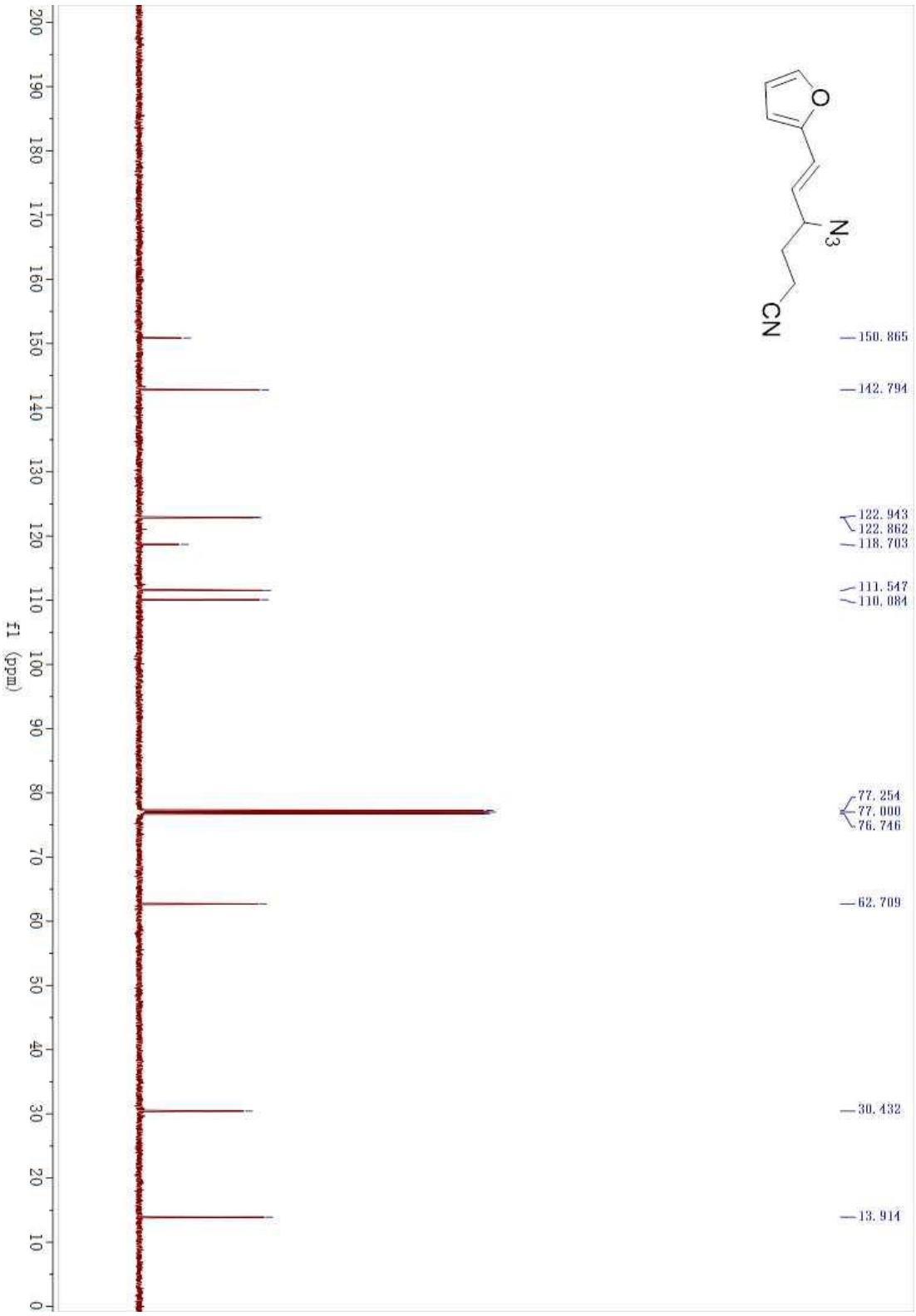
(*E*)-6-(anthracen-9-yl)-4-azidohex-5-enenitrile (3o):



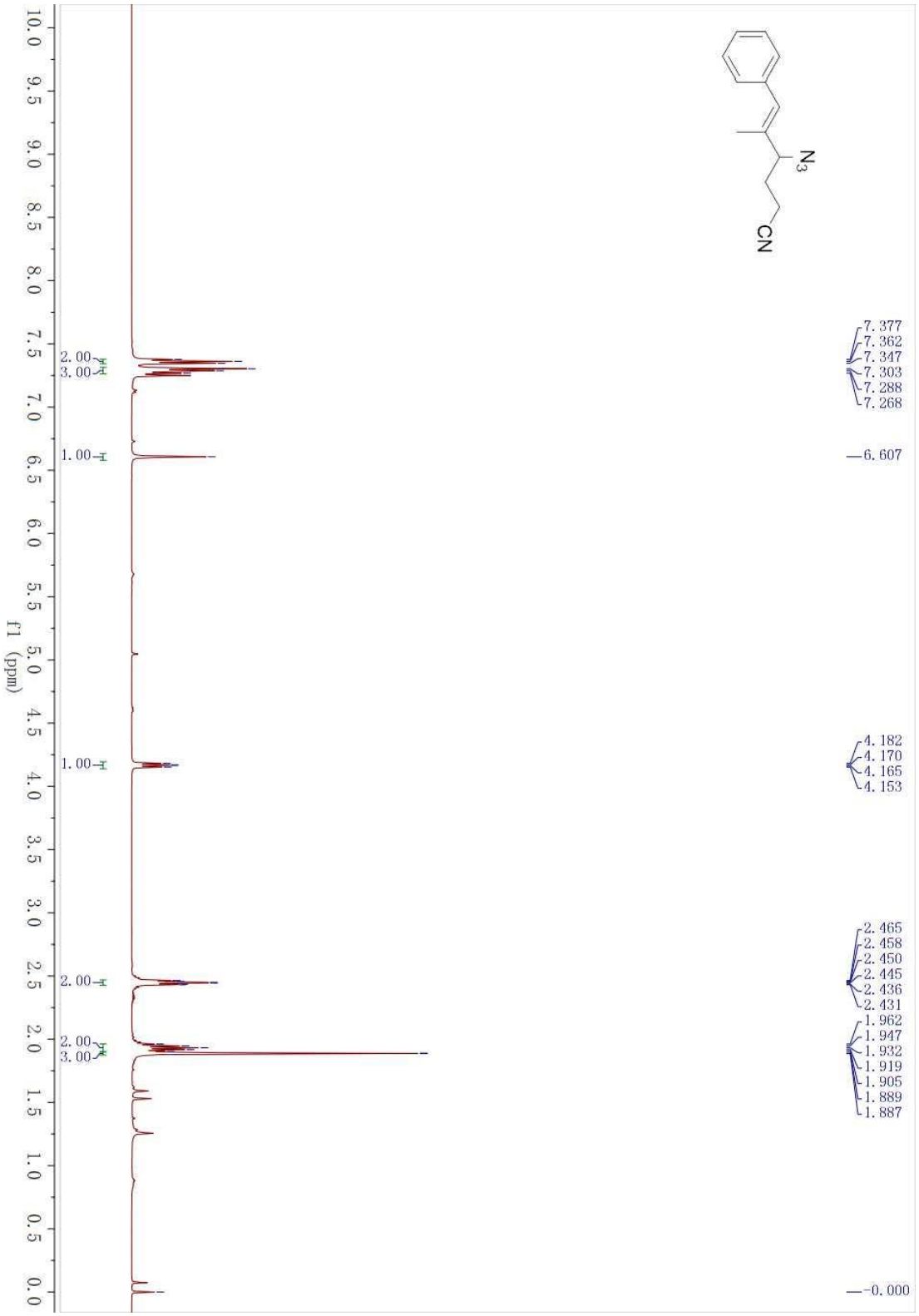

133.724
131.379
131.289
130.426
129.249
128.816
127.223
126.016
125.252
118.740
77.254
77.000
76.746
63.431
30.511
14.994
14.194
f1 (ppm)
N3
CN

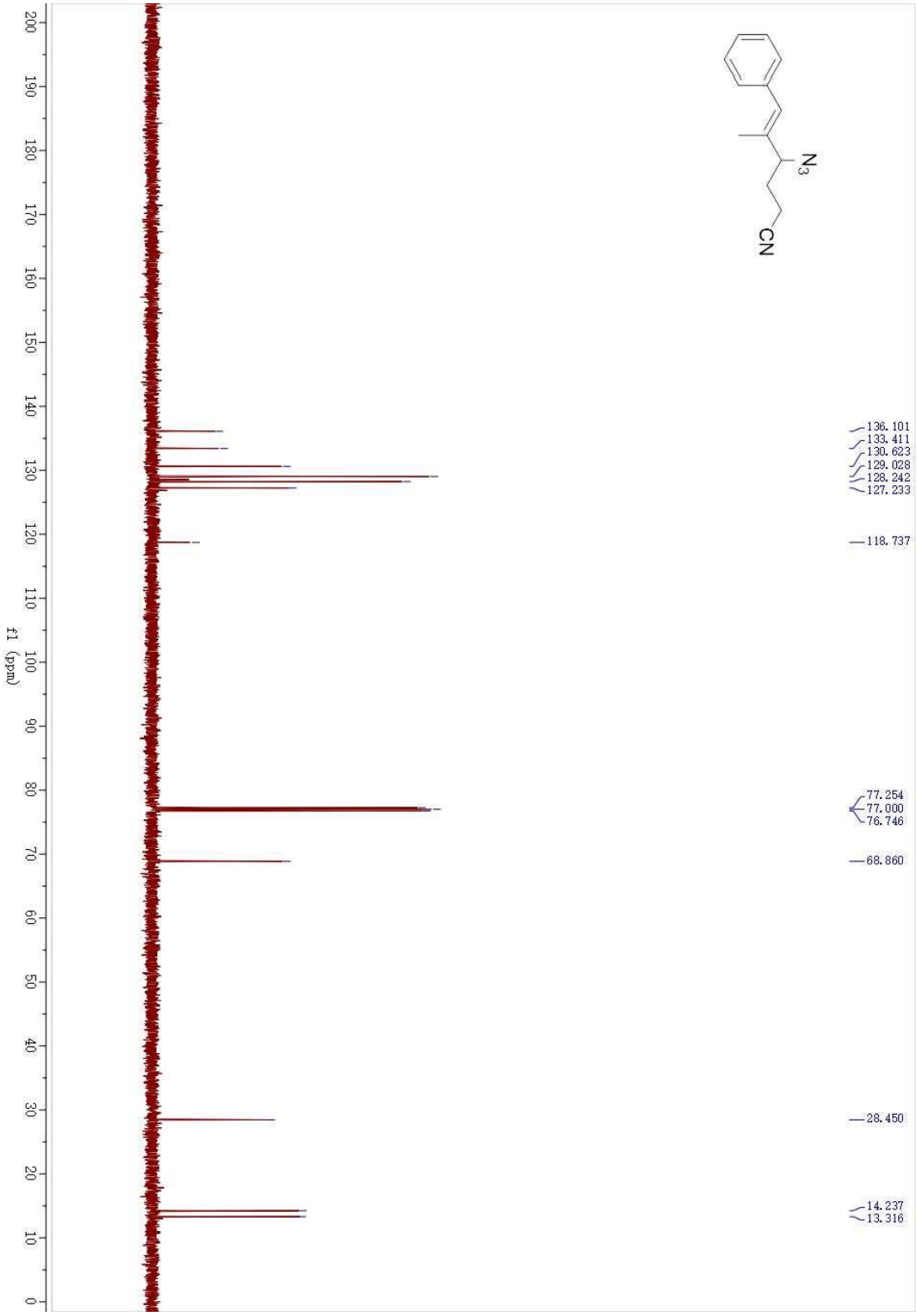
(*E*)-4-azido-6-(furan-2-yl)hex-5-enenitrile (3p):



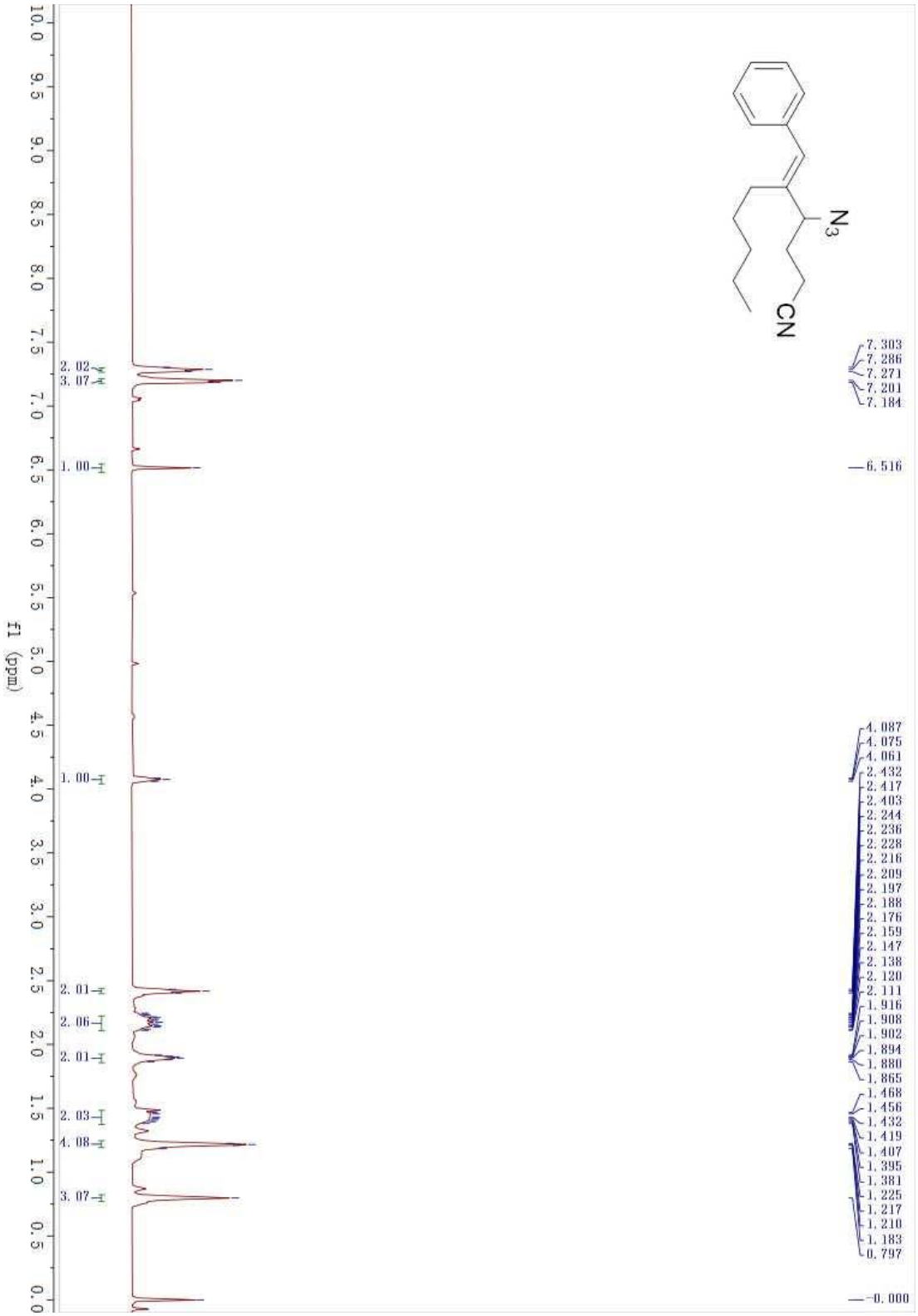

O
N3
CN
150.865
142.794
122.943
122.862
118.703
111.547
110.084
77.254
77.000
76.746
62.709
30.432
13.914
f1 (ppm)

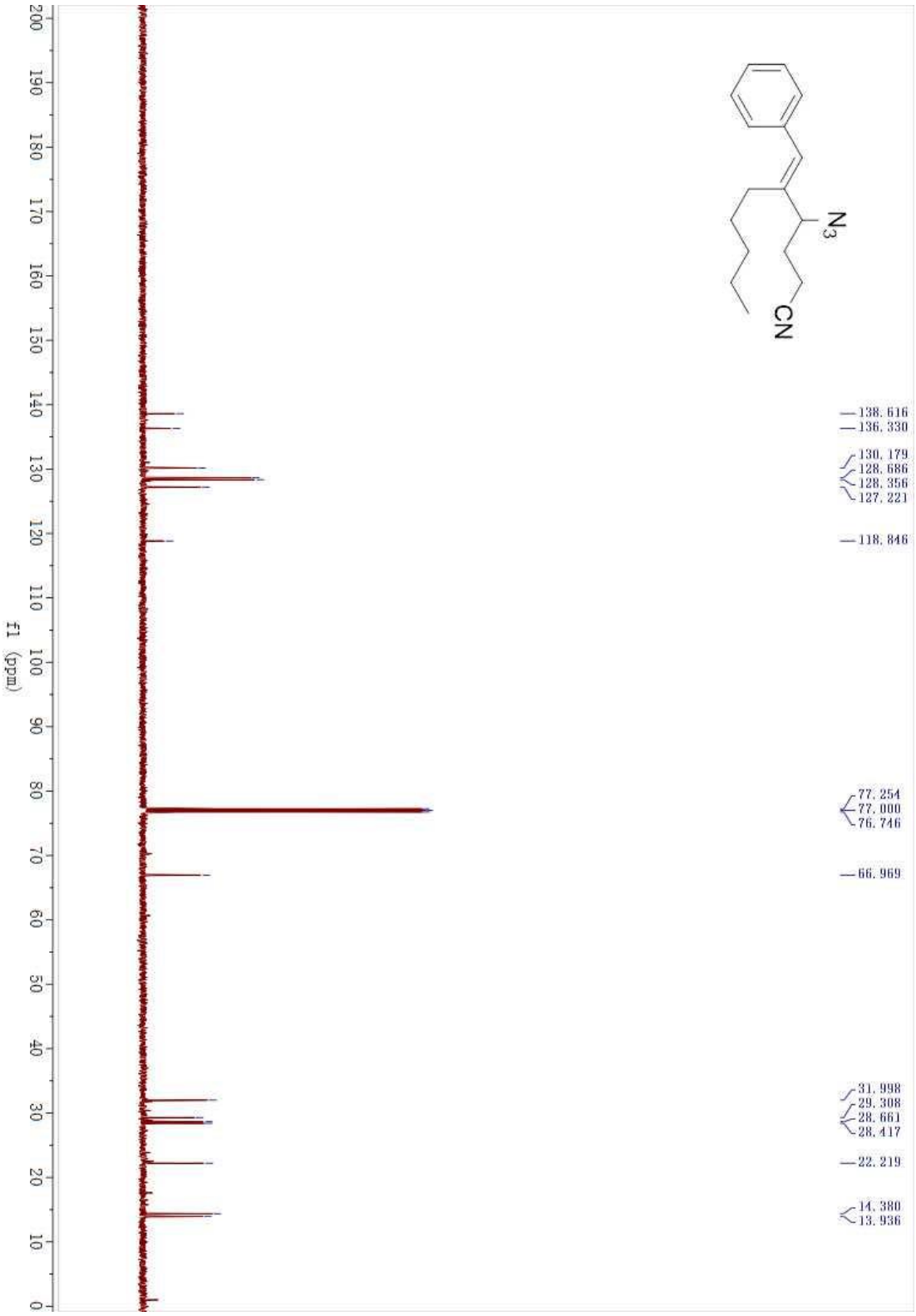
(*E*)-4-azido-5-methyl-6-phenylhex-5-enenitrile (3q):



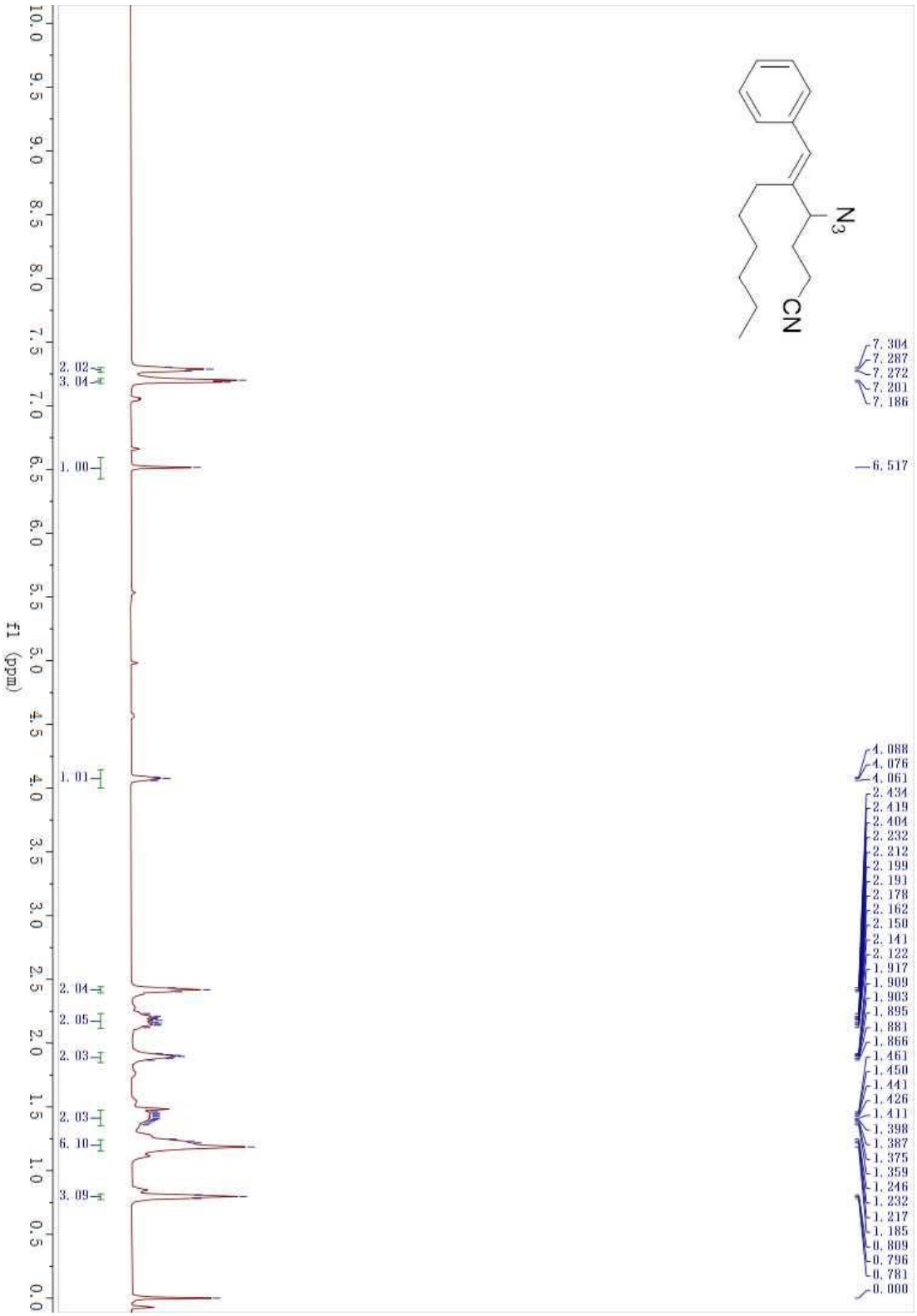

N3
CN
136.101
133.411
130.623
129.028
128.242
127.233
118.737
77.254
77.000
76.746
68.860
28.450
14.237
13.316
f1 (ppm)

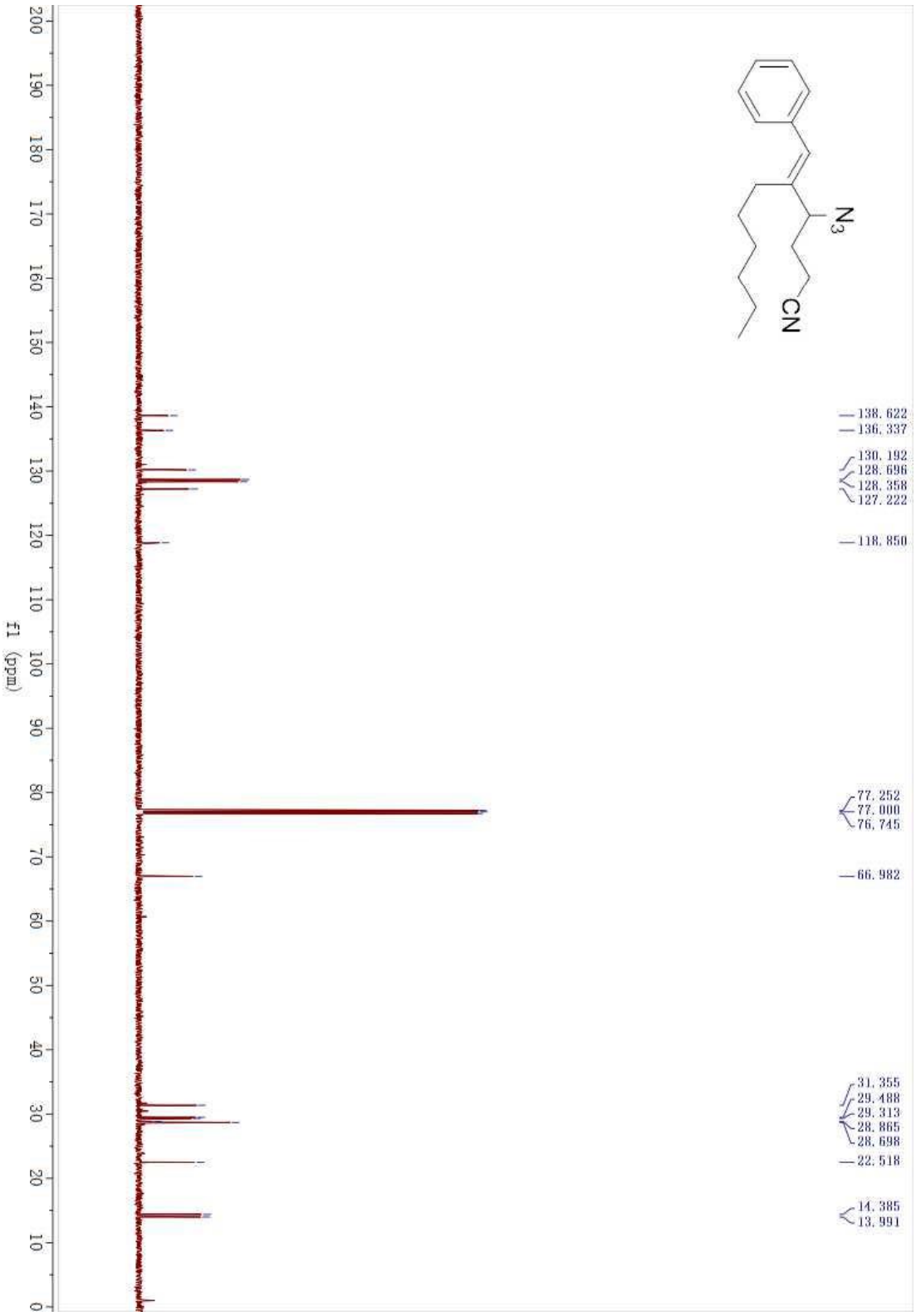
(*E*)-4-azido-5-benzylidenedecanenitrile (3r):



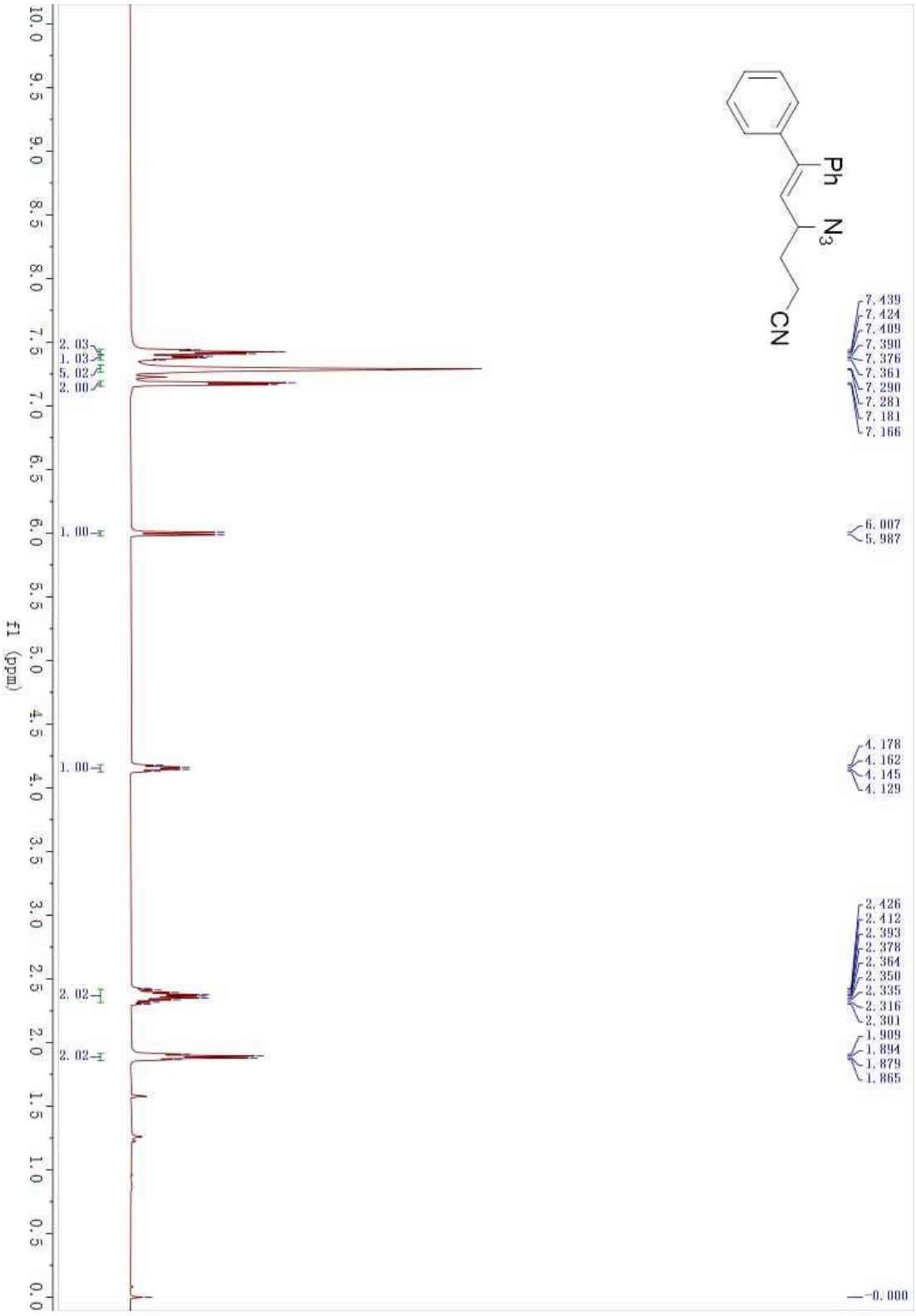

138.616
136.330
130.179
128.686
128.356
127.221
118.846
77.254
77.000
76.746
66.969
31.998
29.308
28.661
28.417
22.219
14.380
13.936
f1 (ppm)
N3
CN

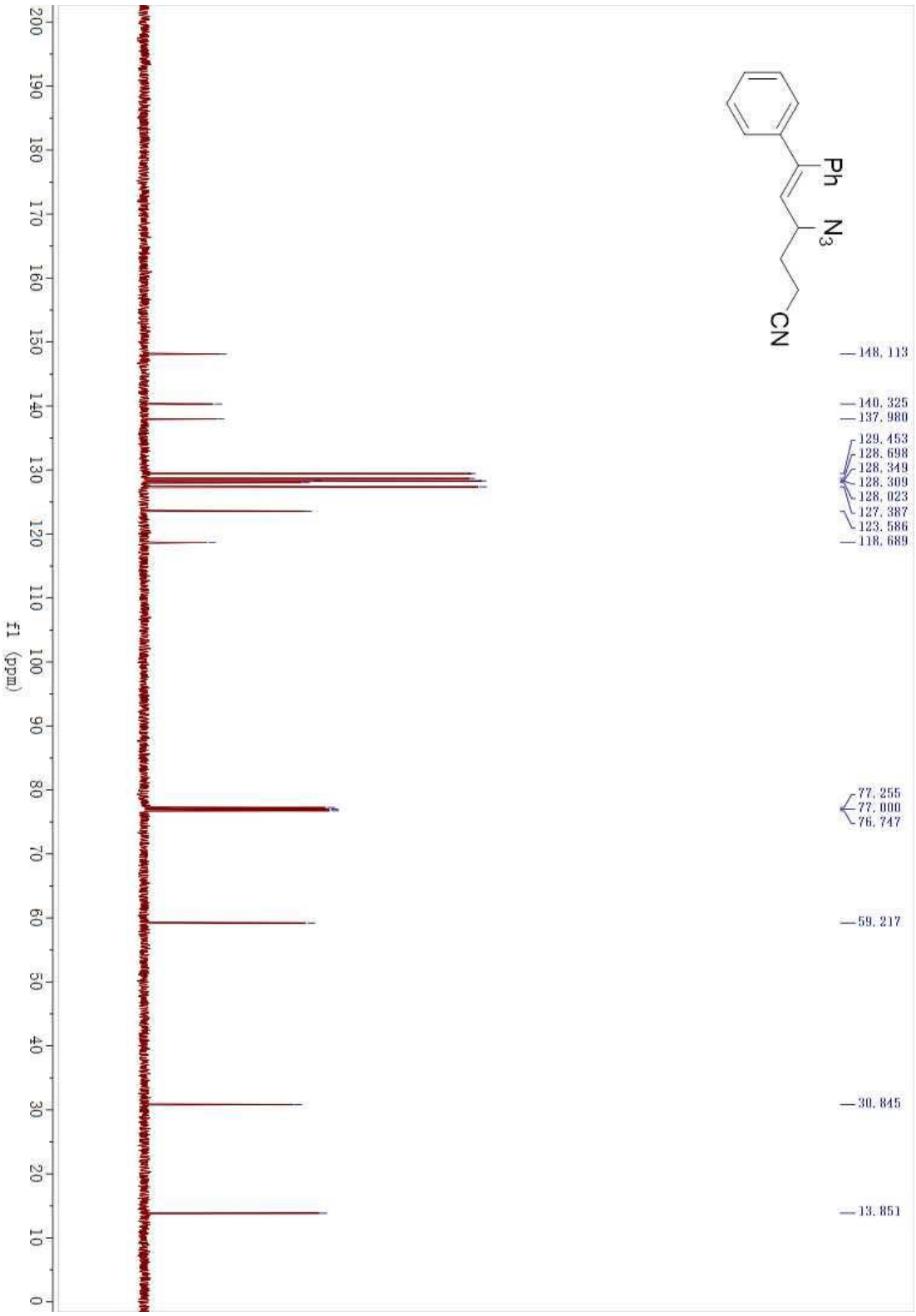
(*E*)-4-azido-5-benzylideneundecanenitrile (3s):



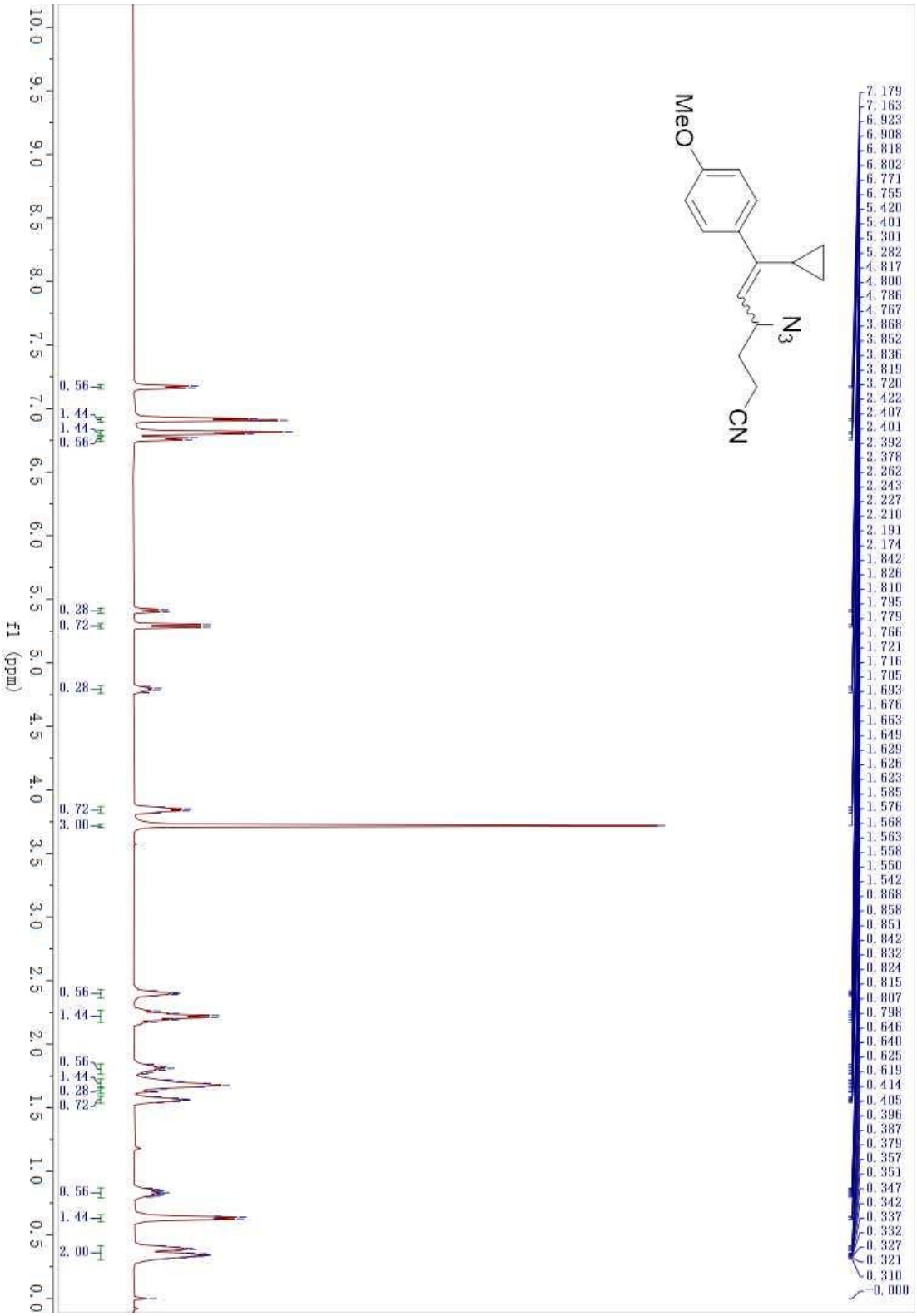

N3
CN
138.622
136.337
130.192
128.696
128.358
127.222
118.850
77.252
77.000
76.745
66.982
31.355
29.488
29.313
28.865
28.698
22.518
14.385
13.991
f1 (ppm)

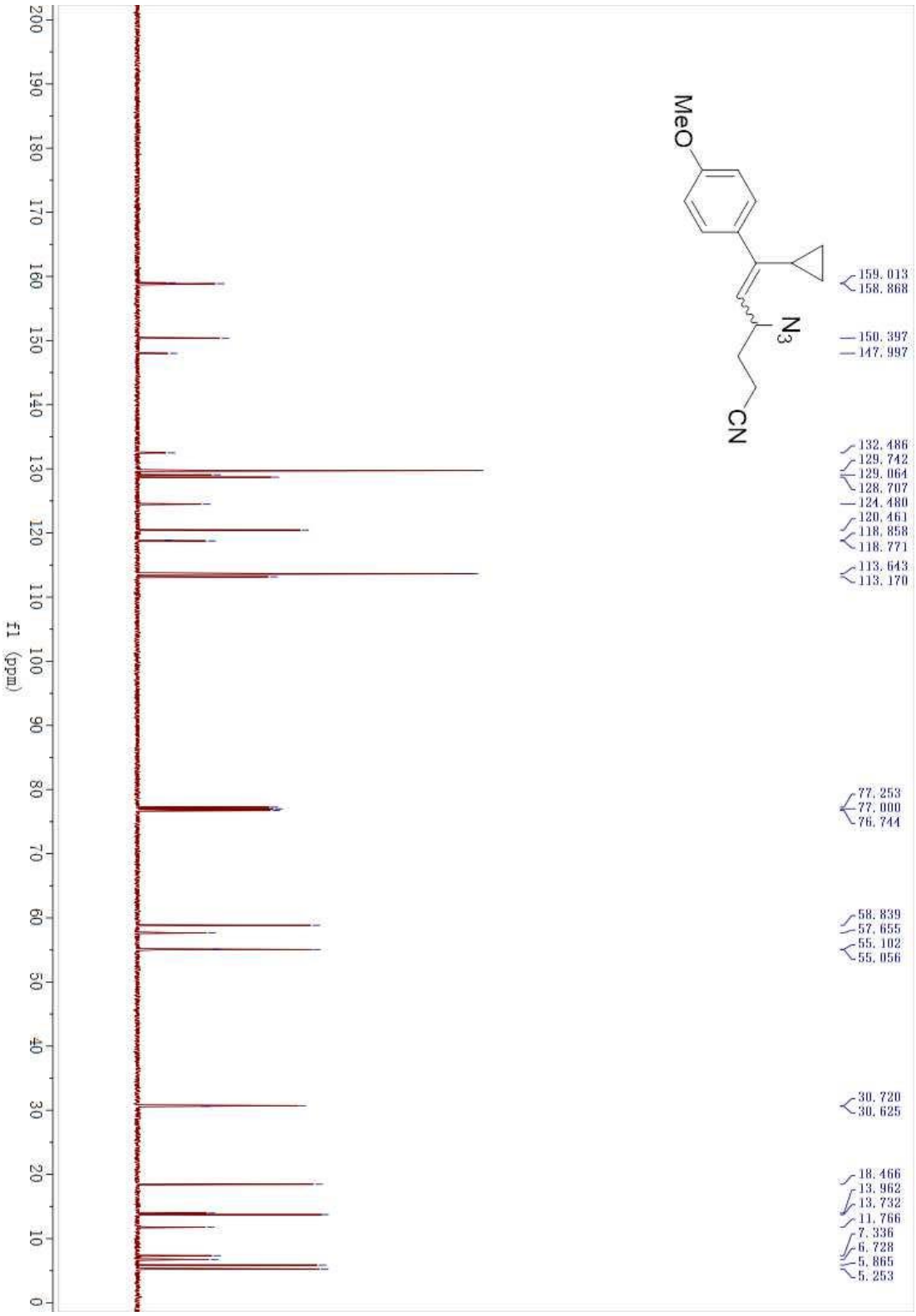
4-azido-6,6-diphenylhex-5-enenitrile (3t):



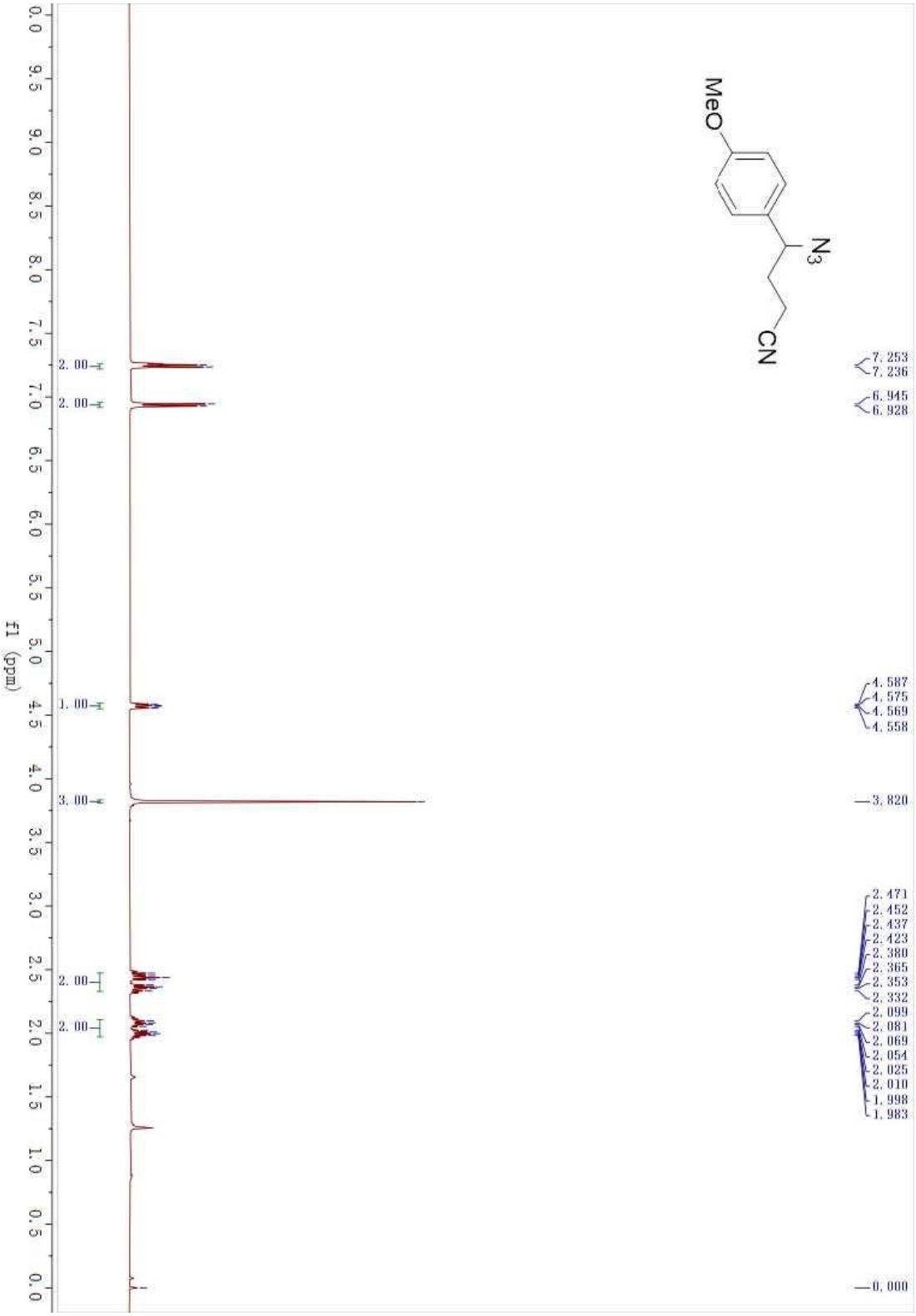

Ph
N3
CN
148.113
140.325
137.980
129.453
128.698
128.349
128.309
128.023
127.387
123.586
118.689
77.255
77.000
76.747
59.217
30.845
13.851
f1 (ppm)

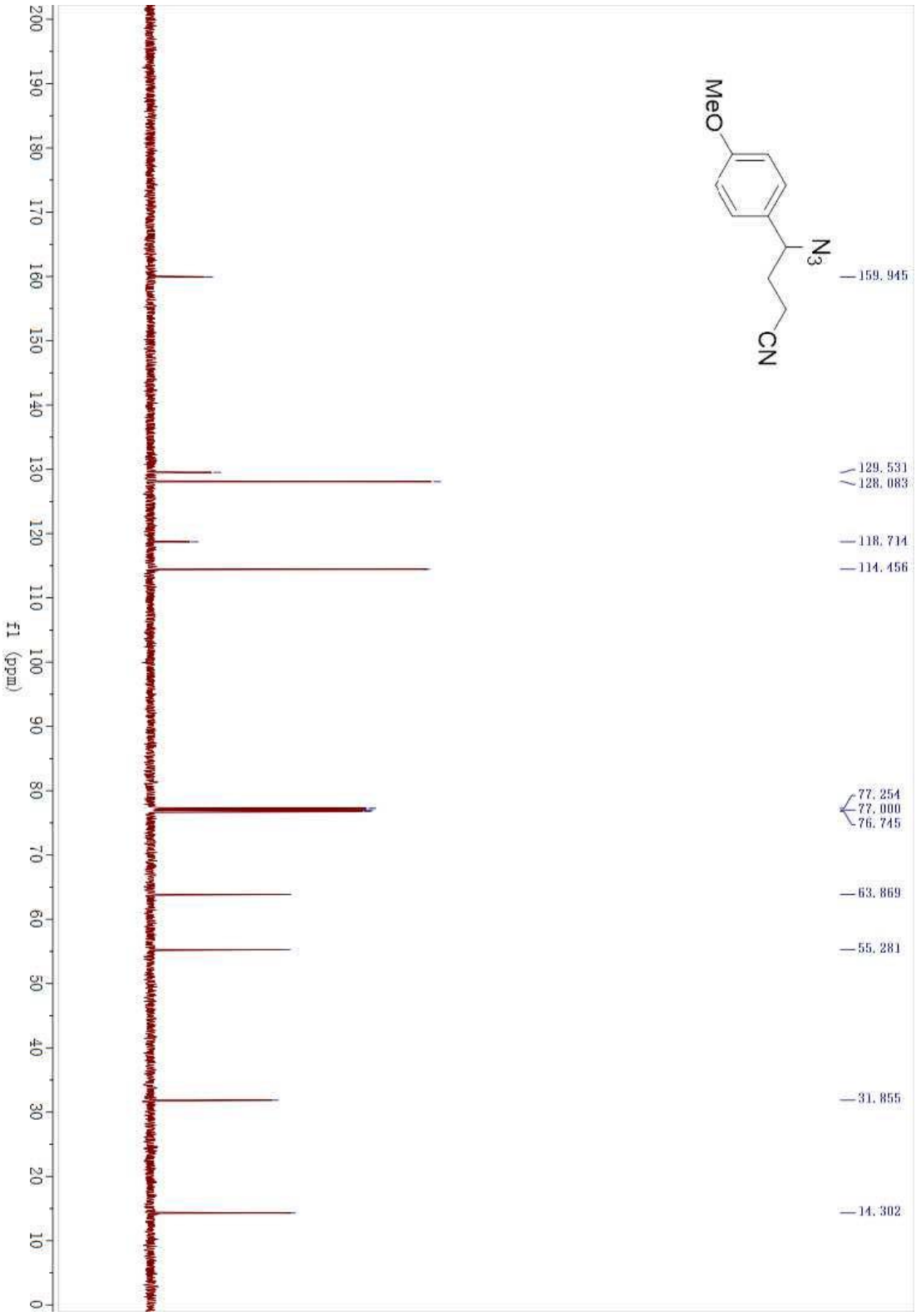
(*E*)-4-azido-6-cyclopropyl-6-(4-methoxyphenyl)hex-5-enenitrile (3u):



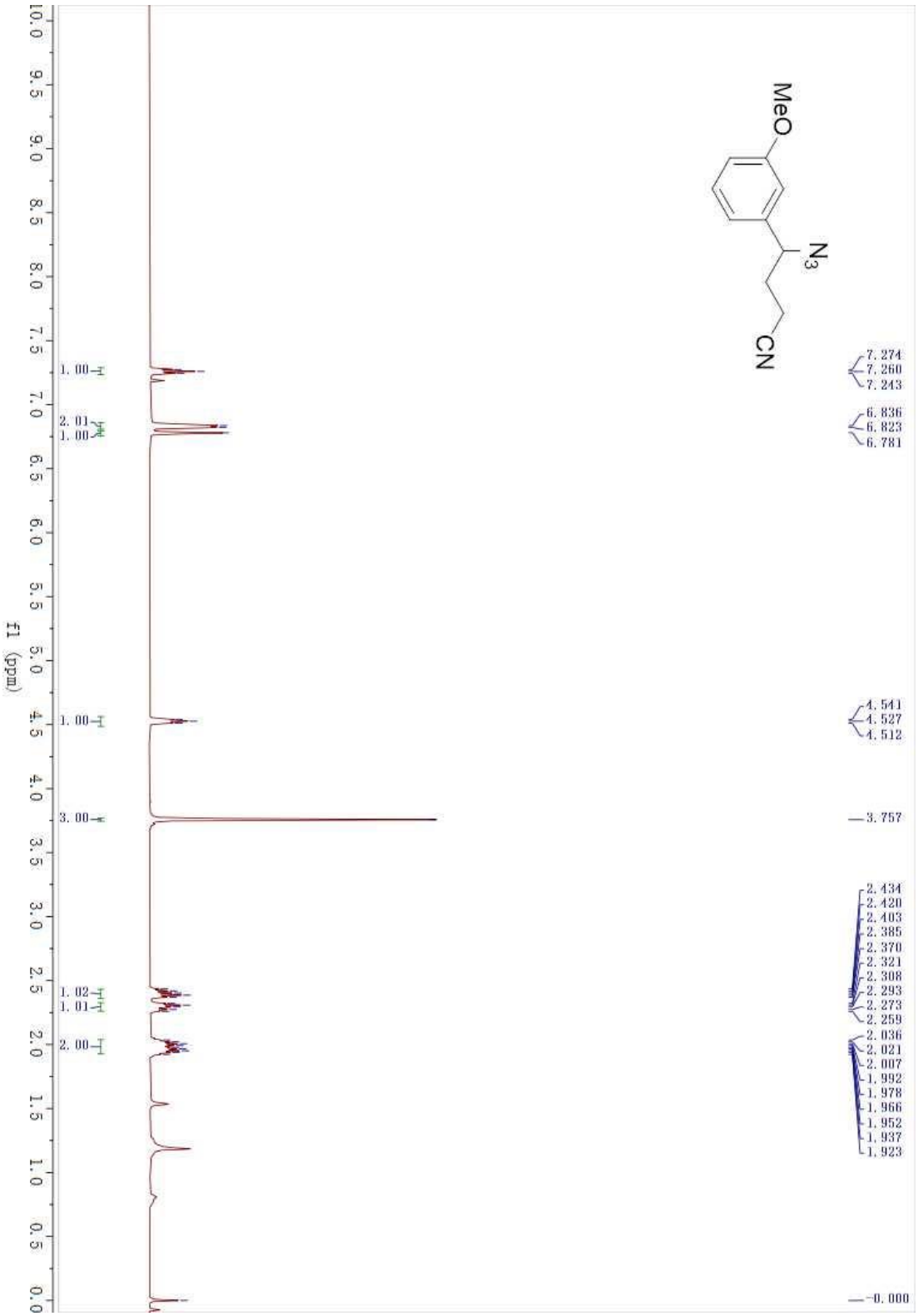

MeO
N3
CN
159.013
158.868
150.397
147.997
132.486
129.742
129.064
128.707
124.480
120.461
118.858
118.771
113.643
113.170
77.253
77.000
76.744
58.839
57.655
55.102
55.056
30.720
30.625
18.466
13.962
13.732
11.766
7.336
6.728
5.865
5.253
f1 (ppm)

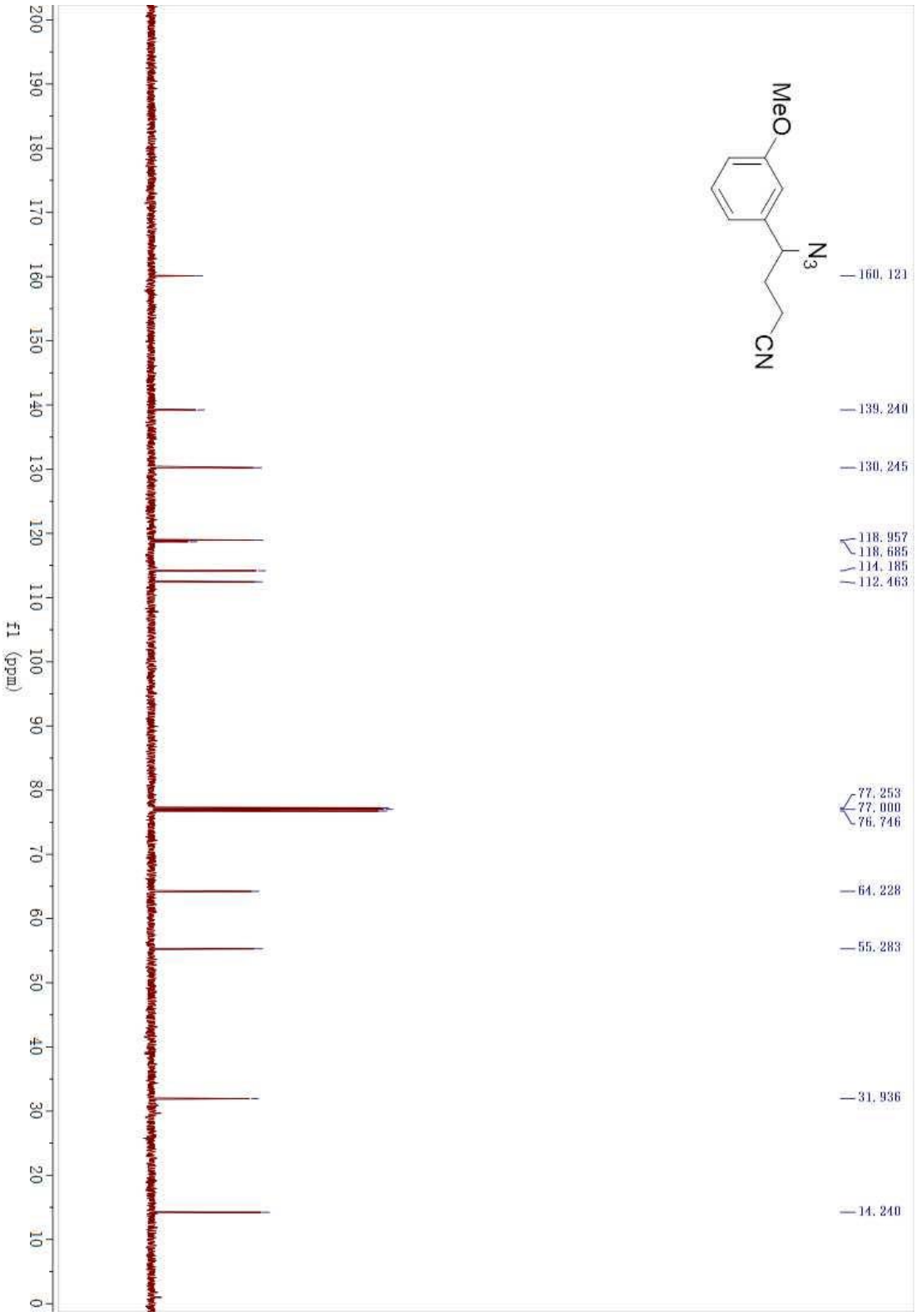
4-azido-4-(4-methoxyphenyl)butanenitrile (3v):



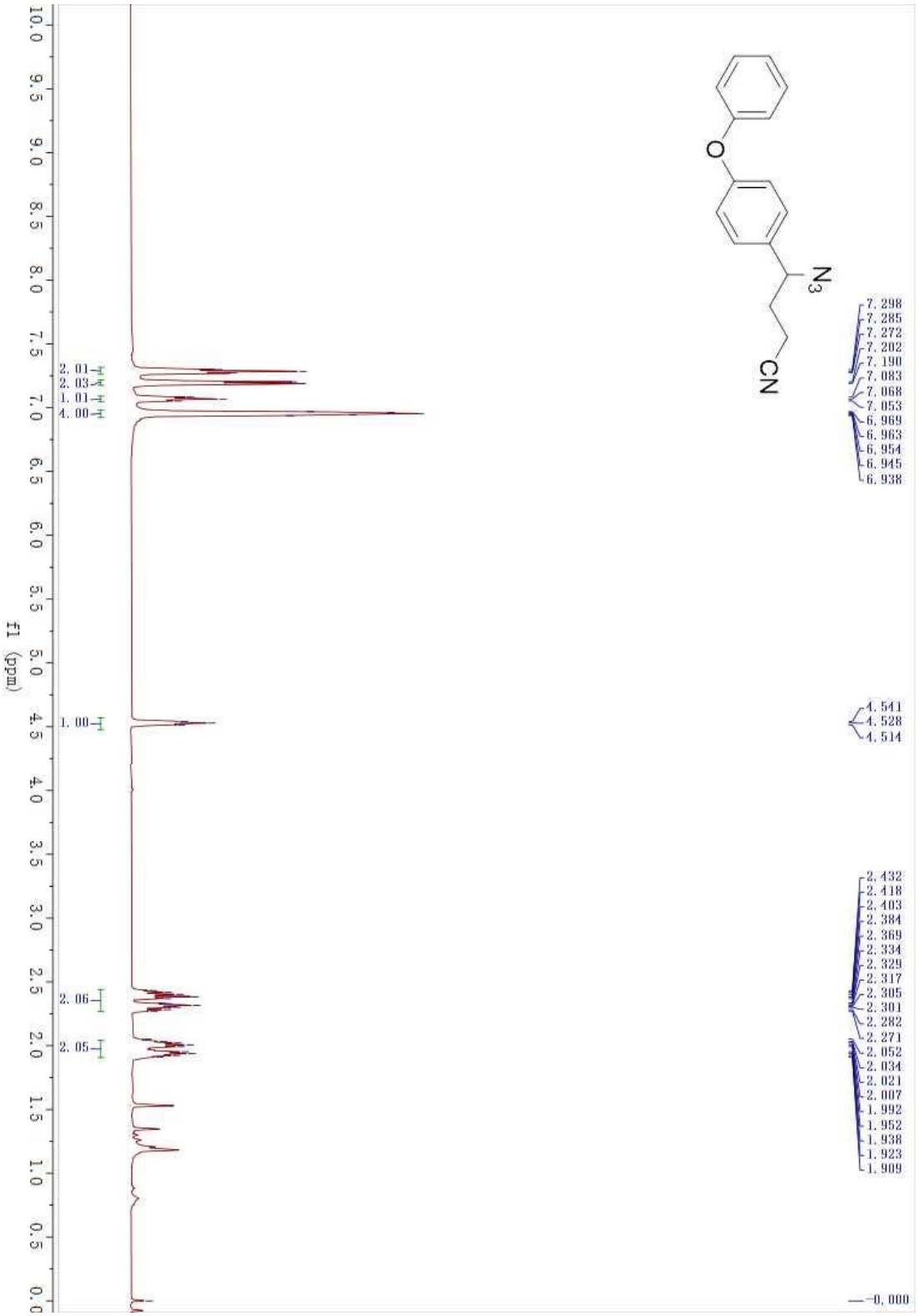

MeO
N3
CN
159.945
129.531
128.083
118.714
114.456
77.254
77.000
76.745
63.869
55.281
31.855
14.302
f1 (ppm)

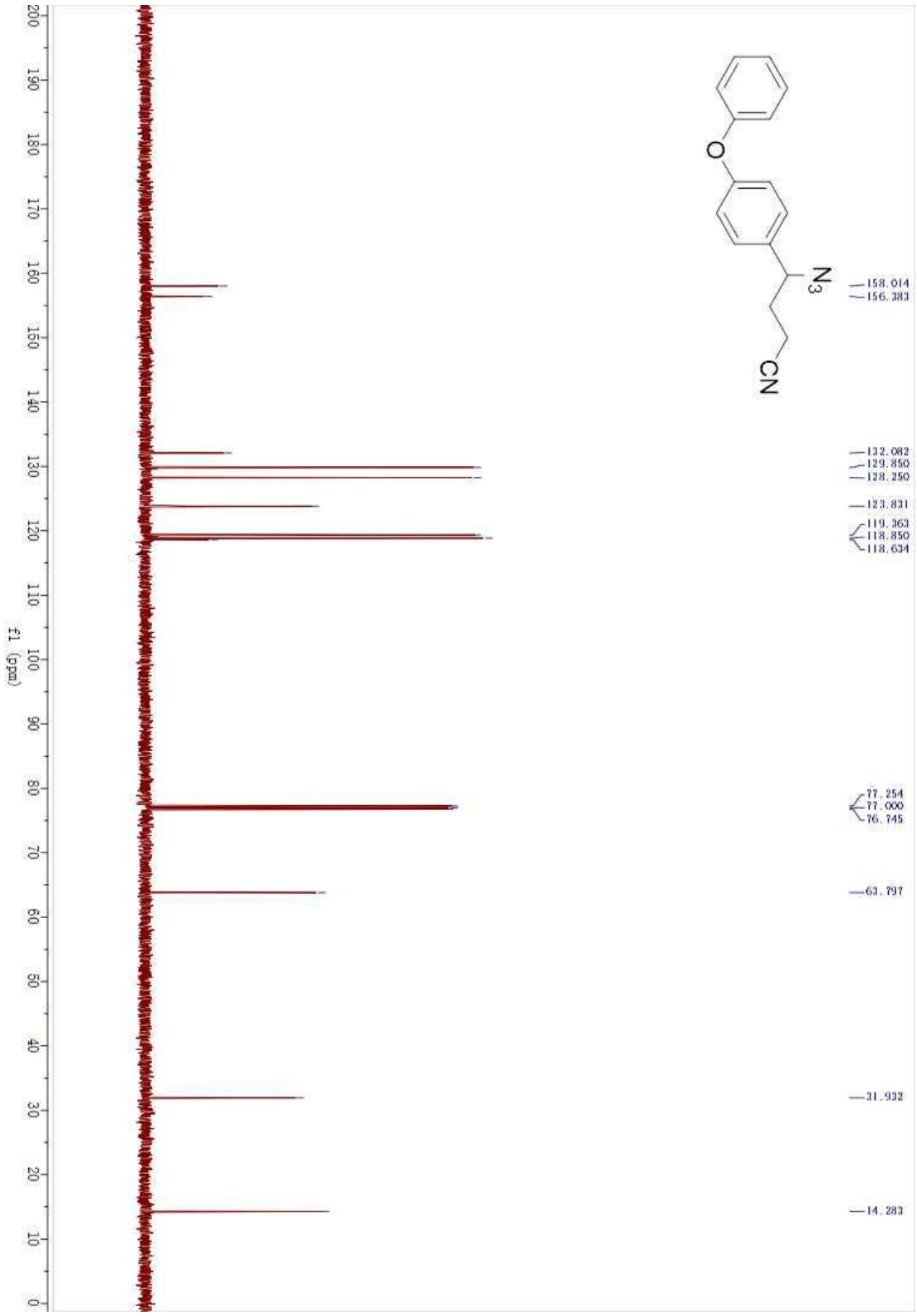
4-azido-4-(3-methoxyphenyl)butanenitrile (3w):



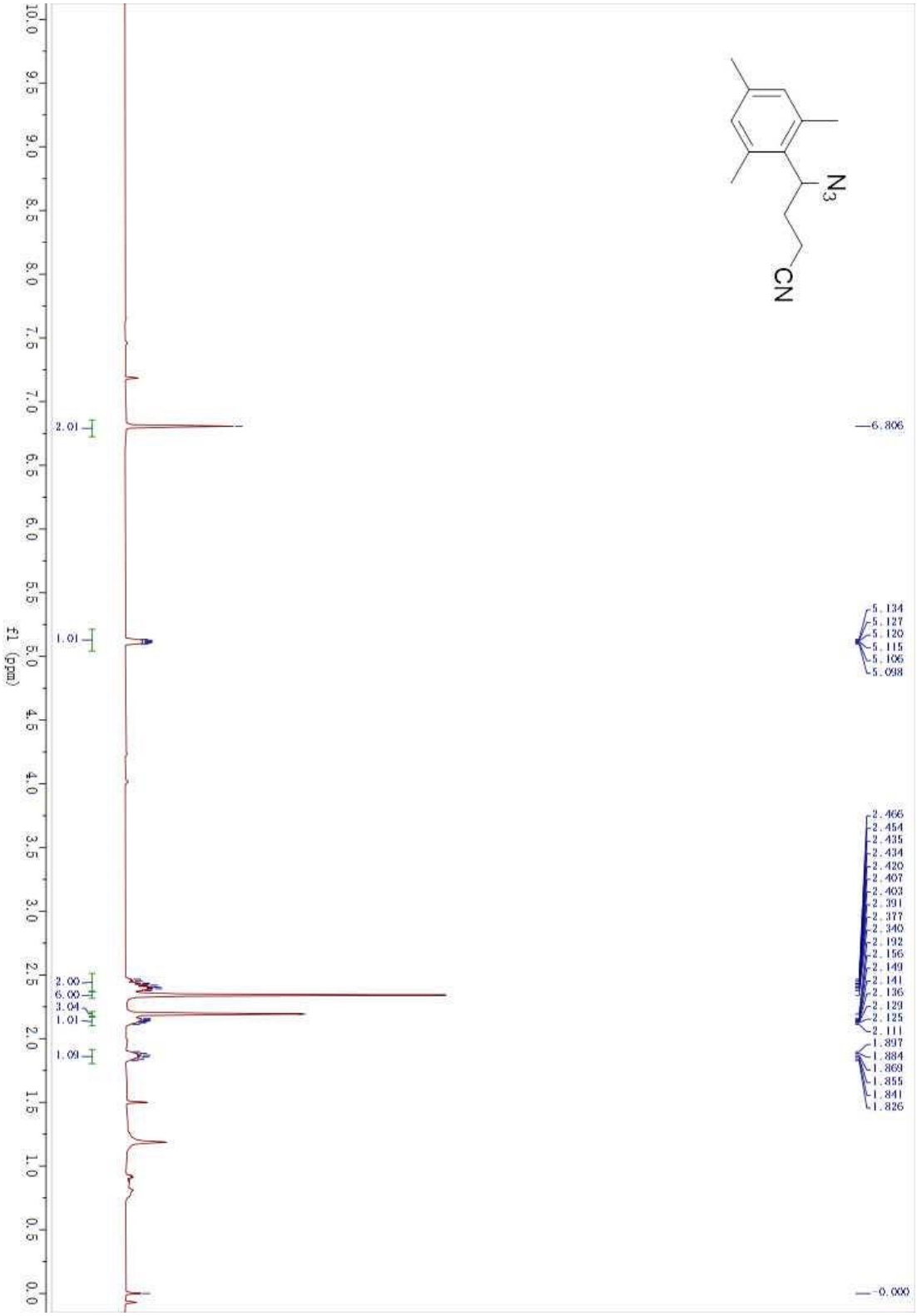

MeO
N3
CN
160.121
139.240
130.245
118.957
118.685
114.185
112.463
77.253
77.000
76.746
64.228
55.283
31.936
14.240
f1 (ppm)

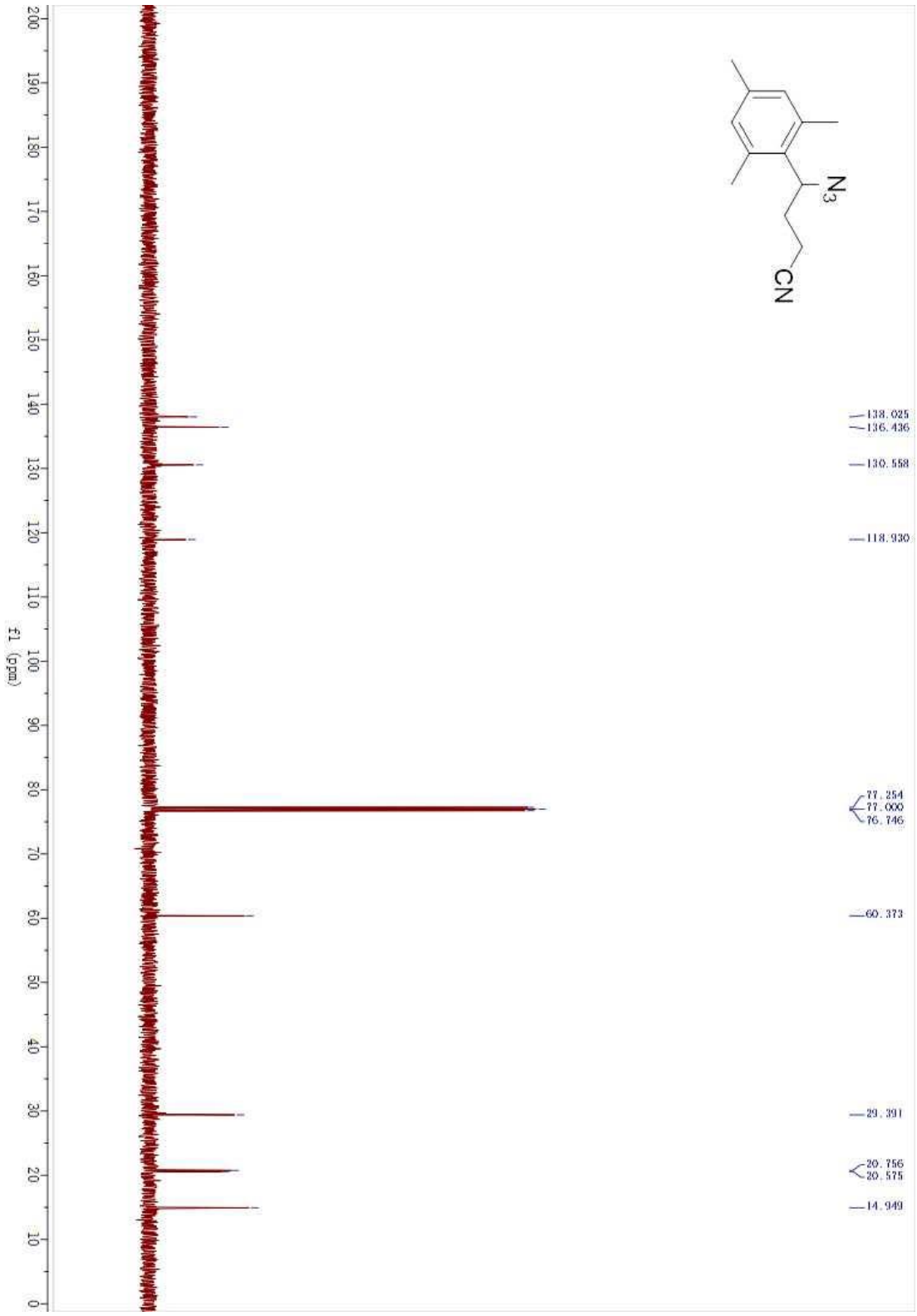
4-azido-4-(4-phenoxyphenyl)butanenitrile (3x):



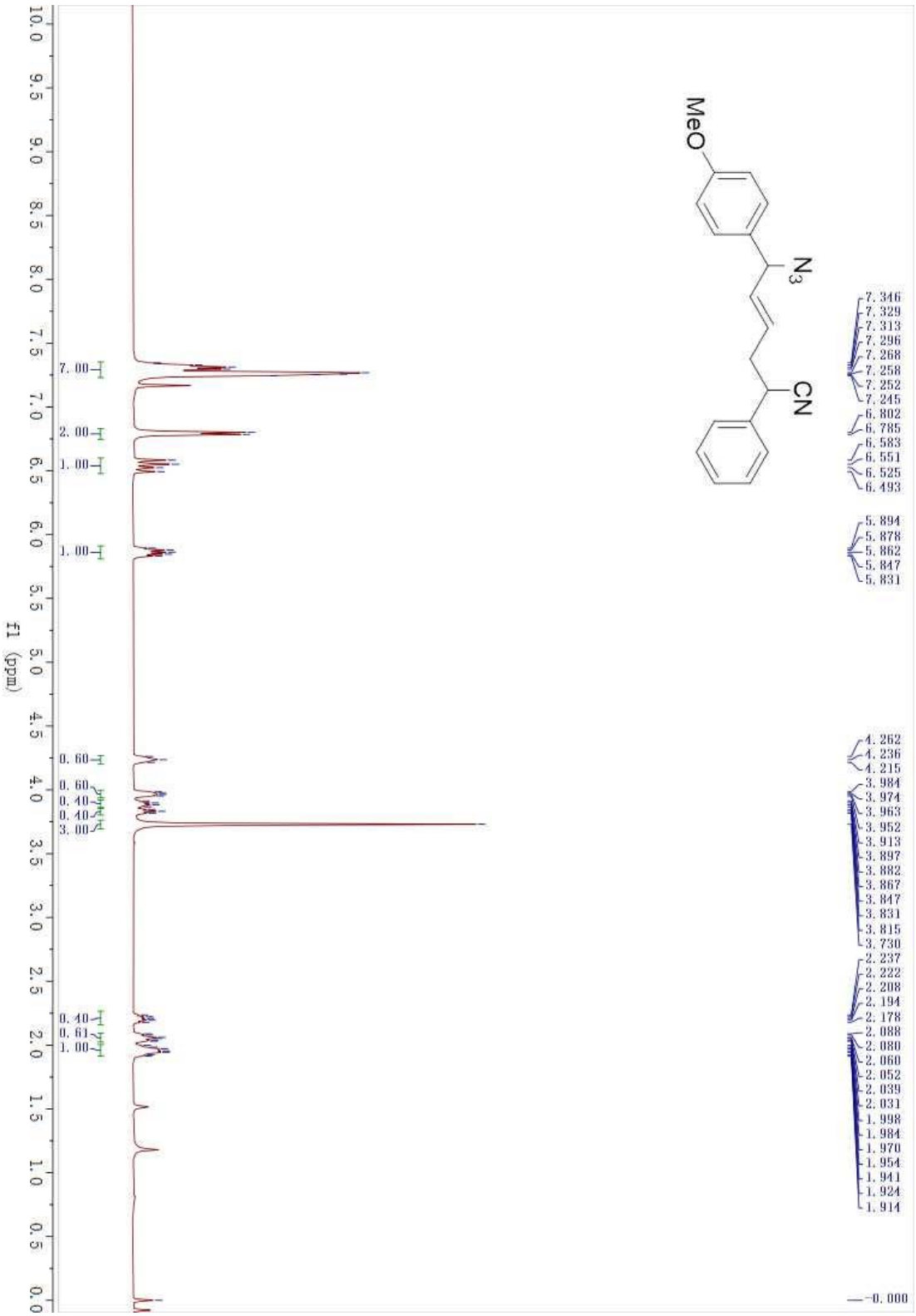

158.014
156.383
132.082
129.850
128.250
123.831
119.363
118.850
118.634
77.254
77.000
76.745
63.797
31.932
14.283
f1 (ppm)
O
N3
CN

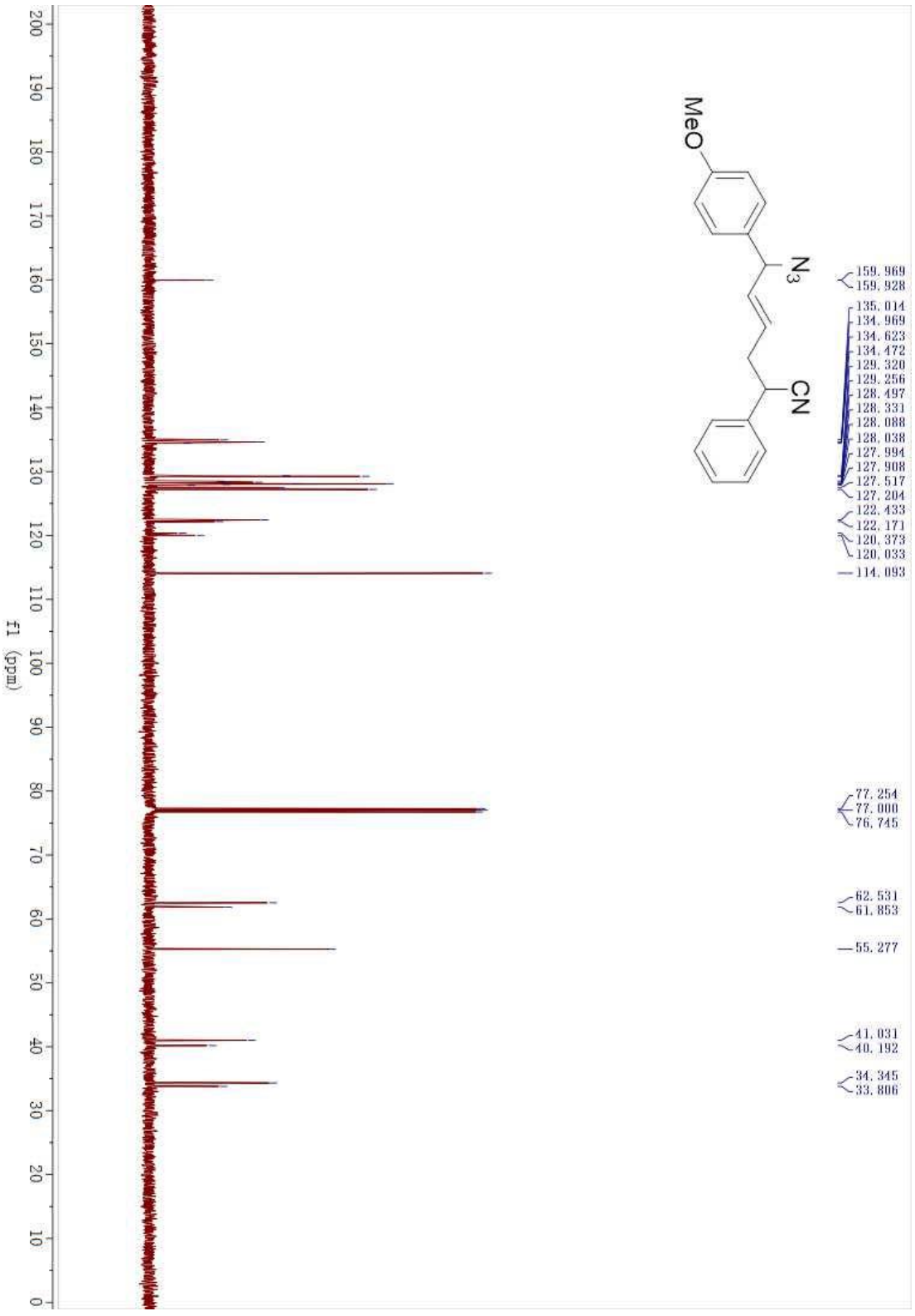
4-azido-4-mesitylbutanenitrile (3y):



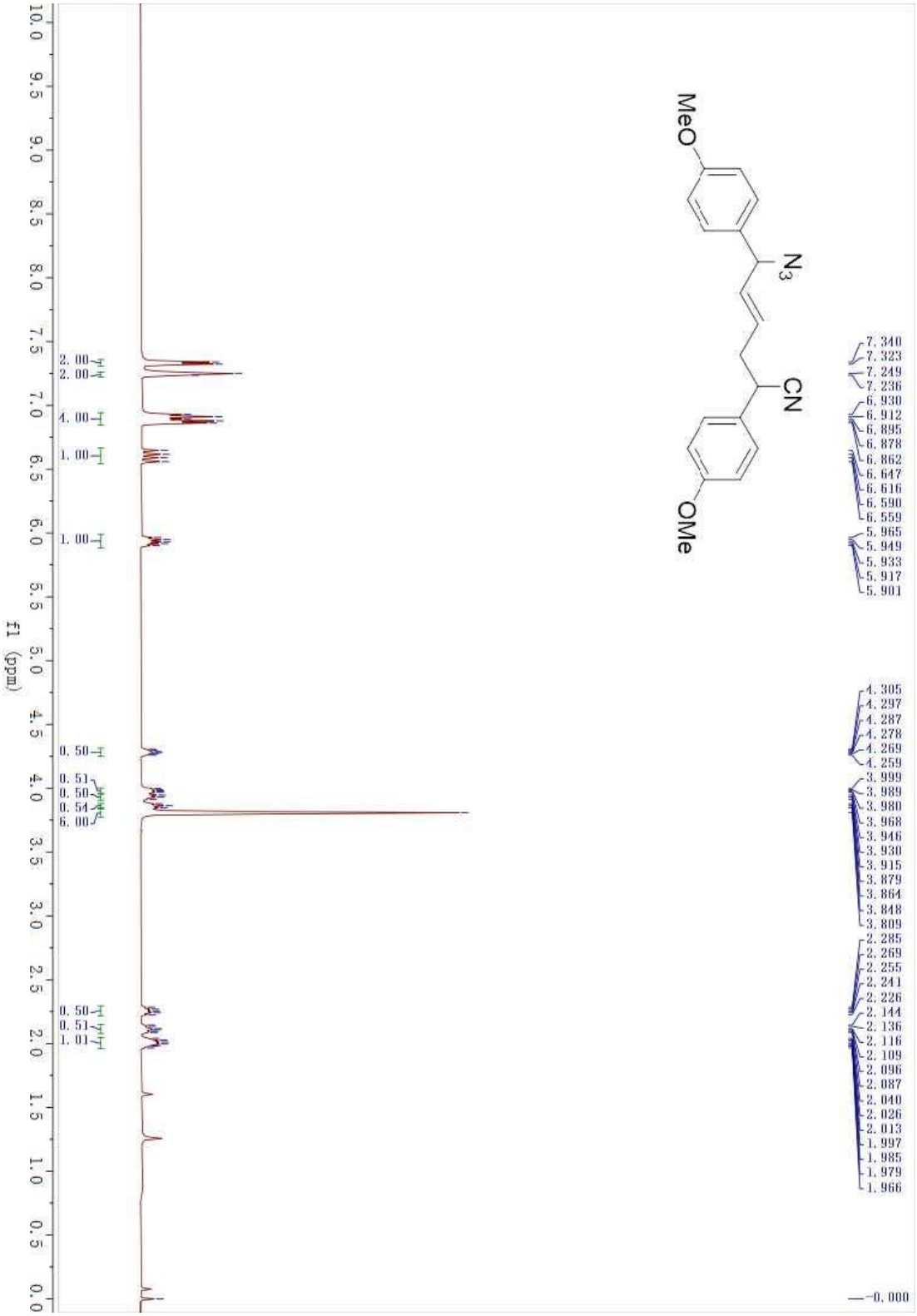

f1 (ppm)
138.025
136.436
130.558
118.930
77.254
77.000
76.746
60.373
29.391
20.756
20.575
14.949
N3
CN

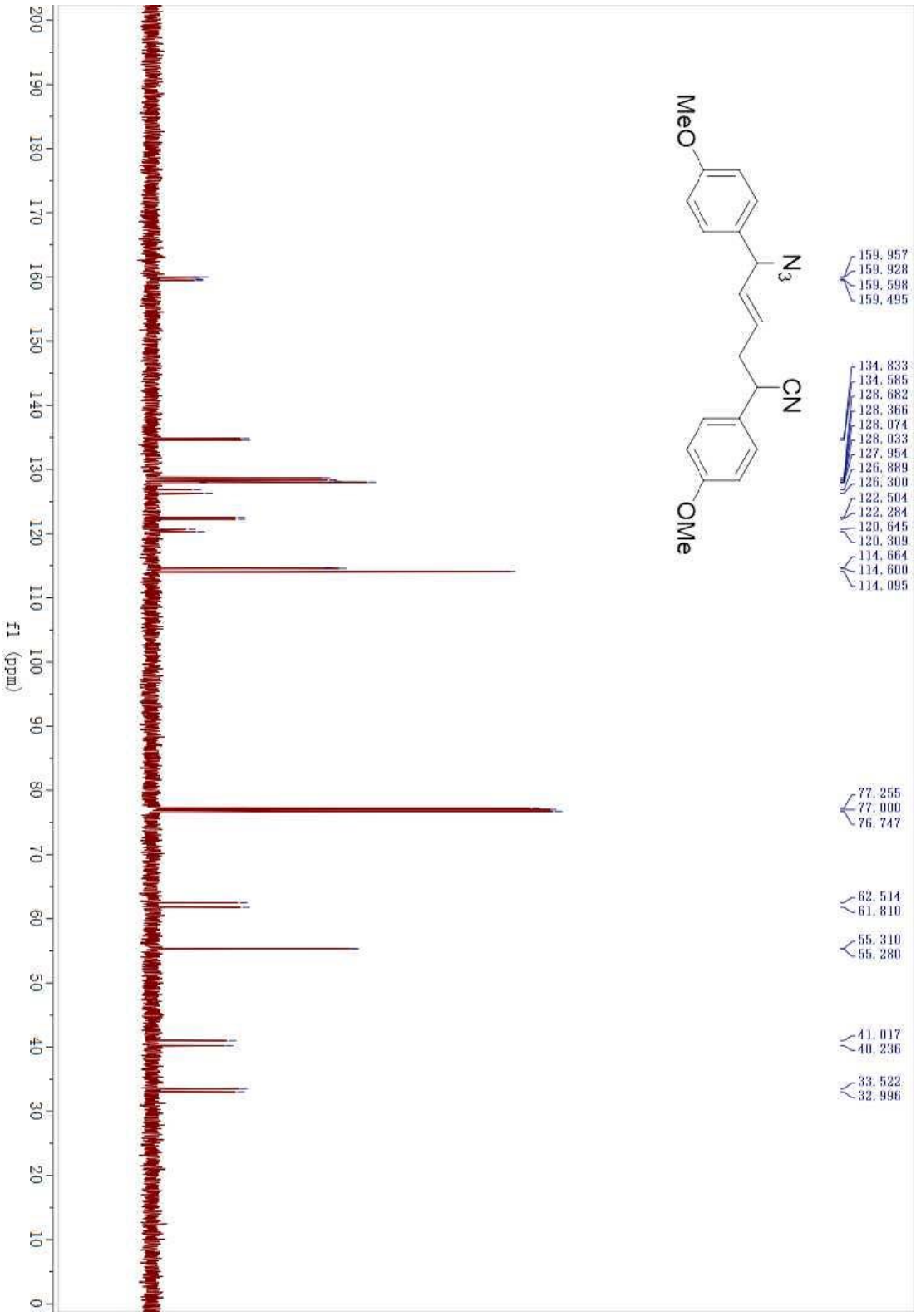
(*E*)-6-azido-6-(4-methoxyphenyl)-2-phenylhex-4-enenitrile (5a):



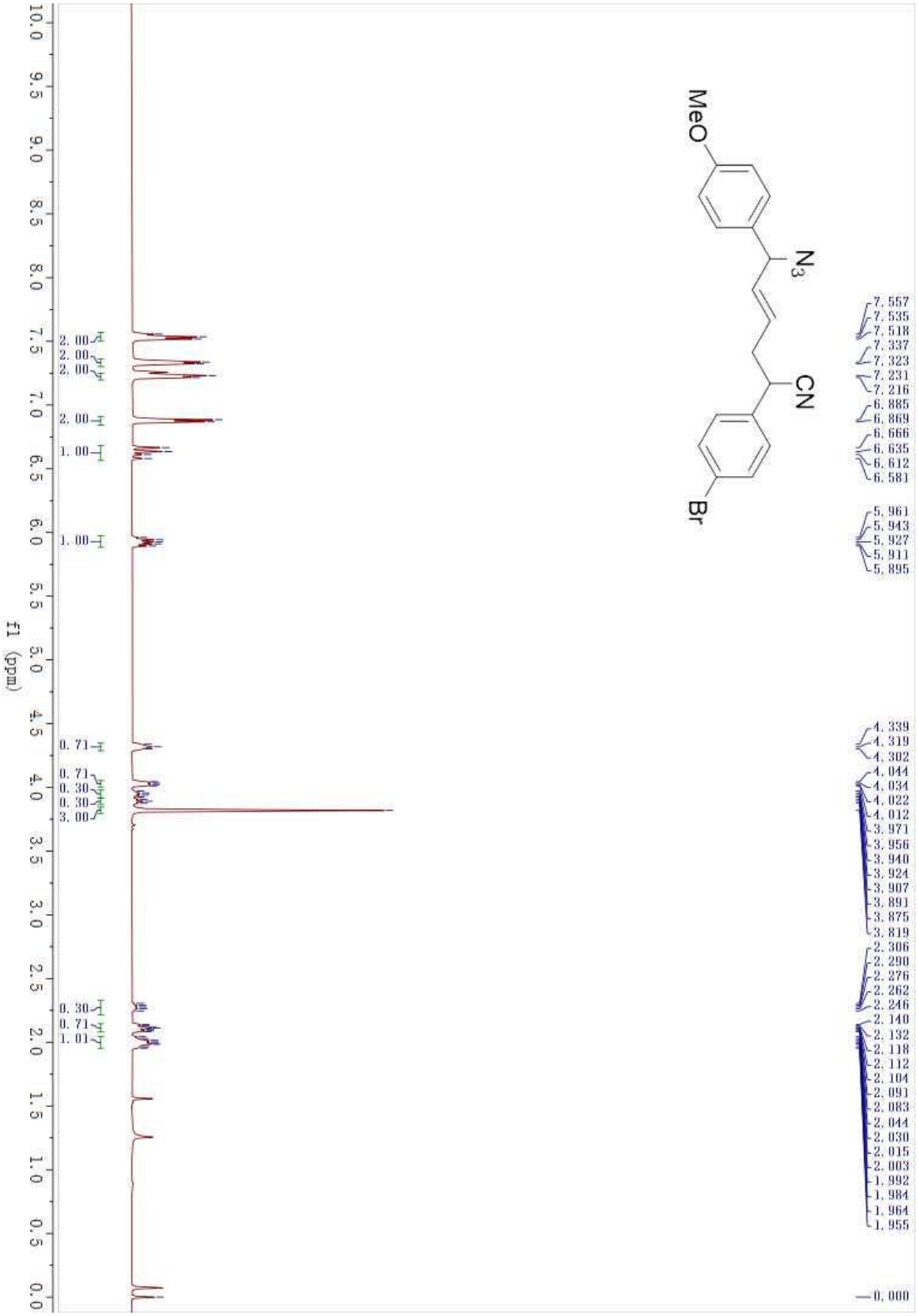

MeO
N3
CN
159.969
159.928
135.014
134.969
134.623
134.472
129.320
129.256
128.497
128.331
128.088
128.038
127.994
127.908
127.517
127.204
122.433
122.171
120.373
120.033
114.093
77.254
77.000
76.745
62.531
61.853
55.277
41.031
40.192
34.345
33.806
f1 (ppm)

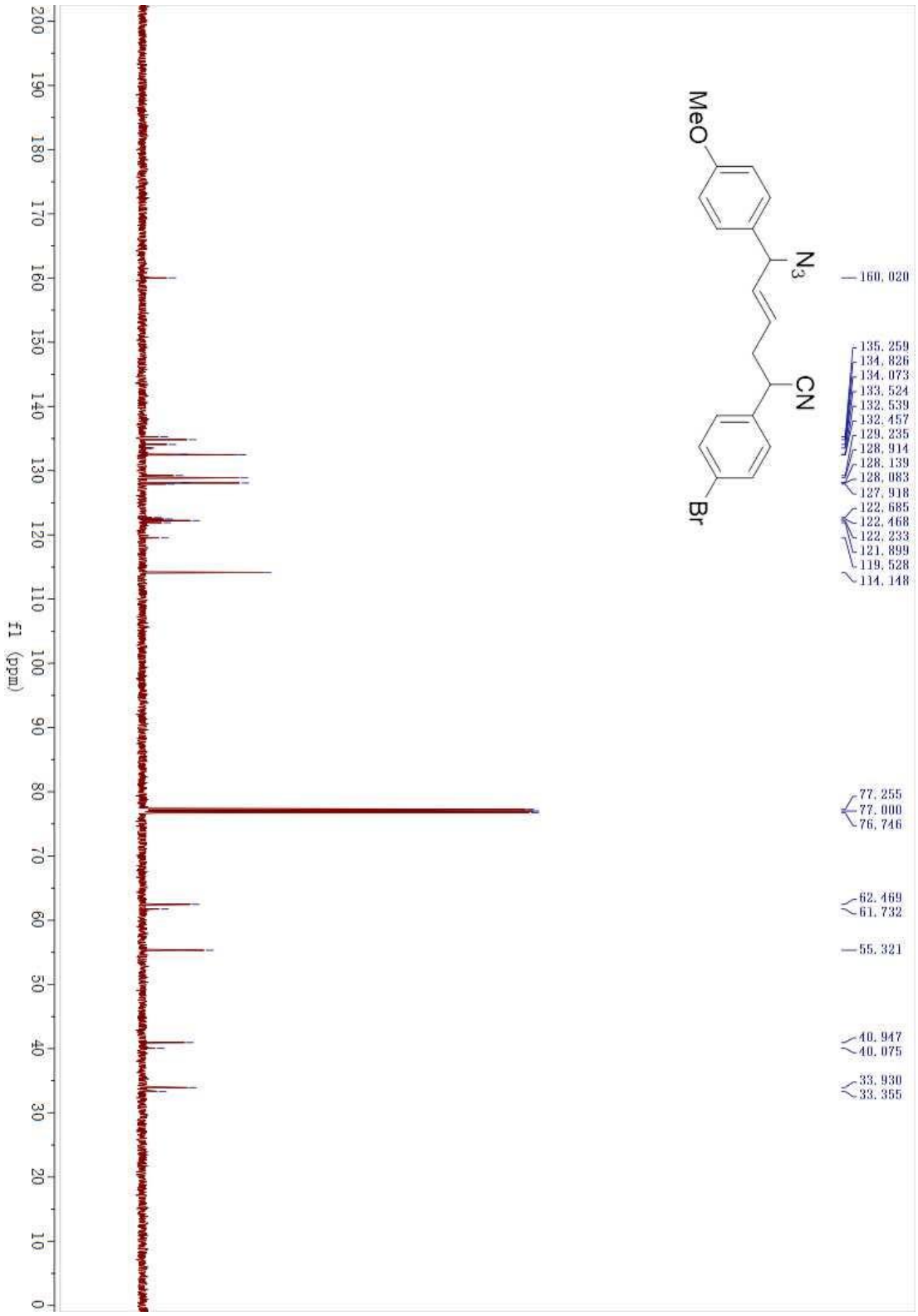
(*E*)-6-azido-2,6-bis(4-methoxyphenyl)hex-4-enenitrile (5b):



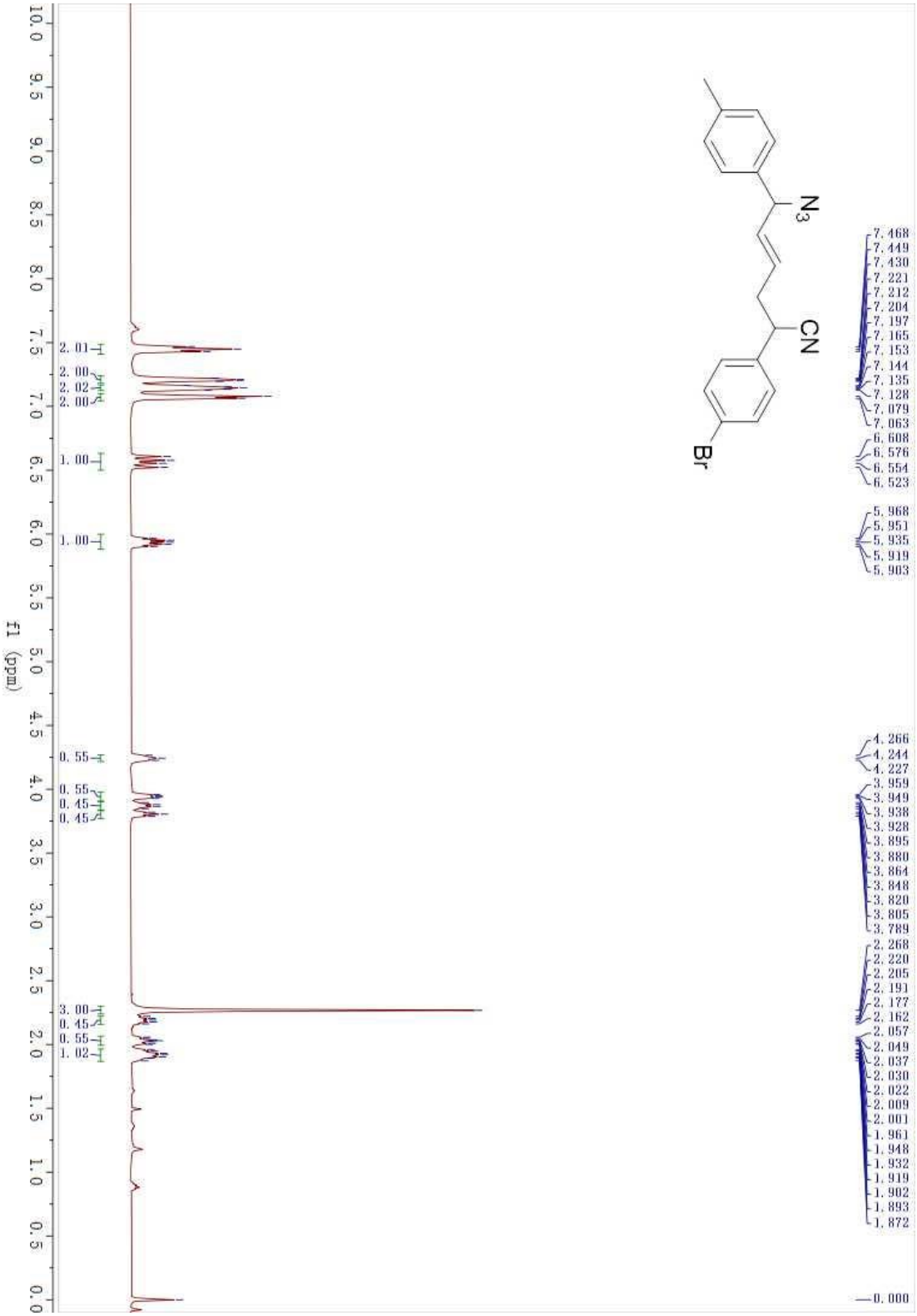

MeO
N3
CN
OMe
159.957
159.928
159.598
159.495
134.833
134.585
128.682
128.366
128.074
128.033
127.954
126.889
126.300
122.504
122.284
120.645
120.309
114.664
114.600
114.095
77.255
77.000
76.747
62.514
61.810
55.310
55.280
41.017
40.236
33.522
32.996
f1 (ppm)

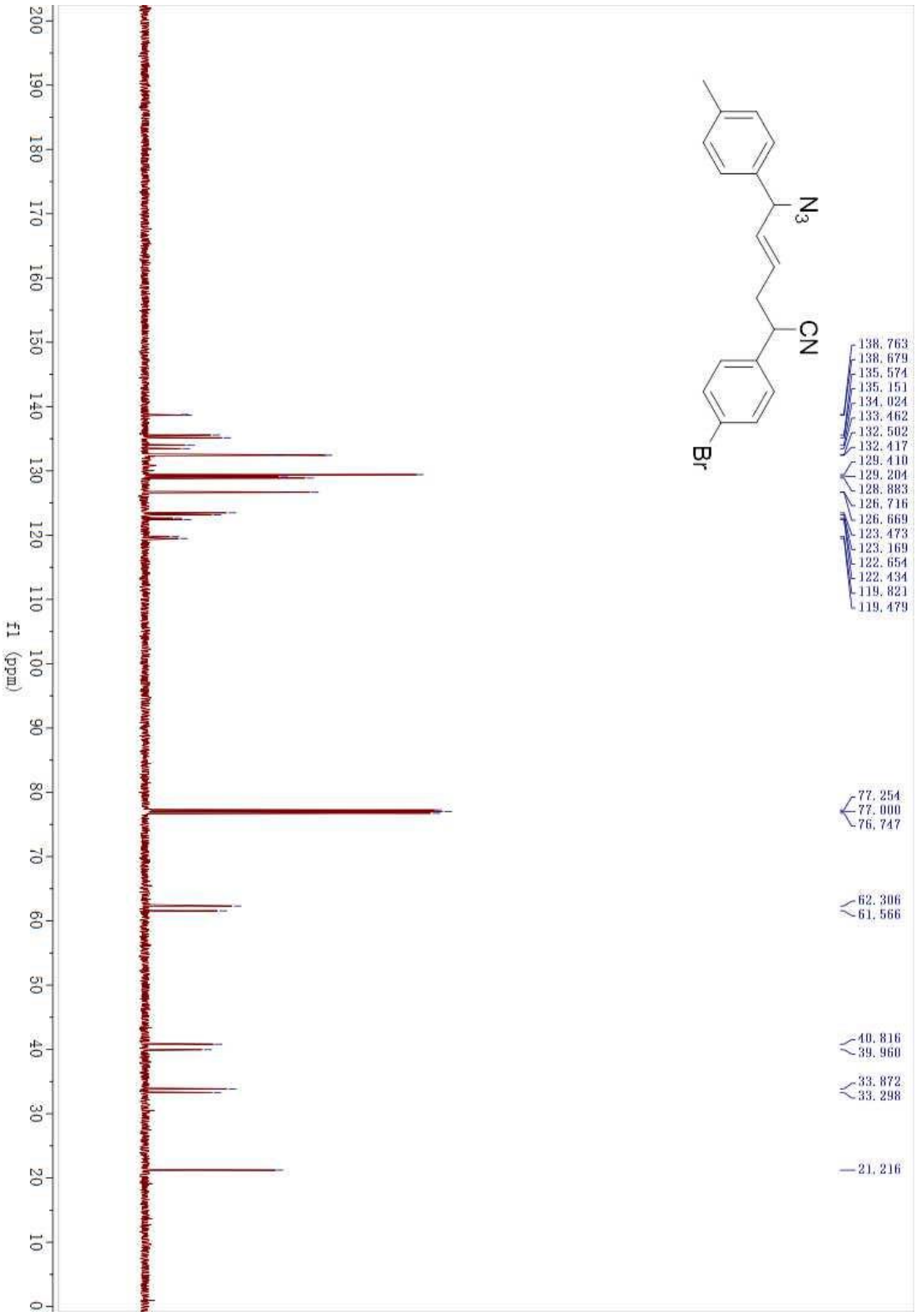
(*E*)-6-azido-2-(4-bromophenyl)-6-(4-methoxyphenyl)hex-4-enenitrile (5c):



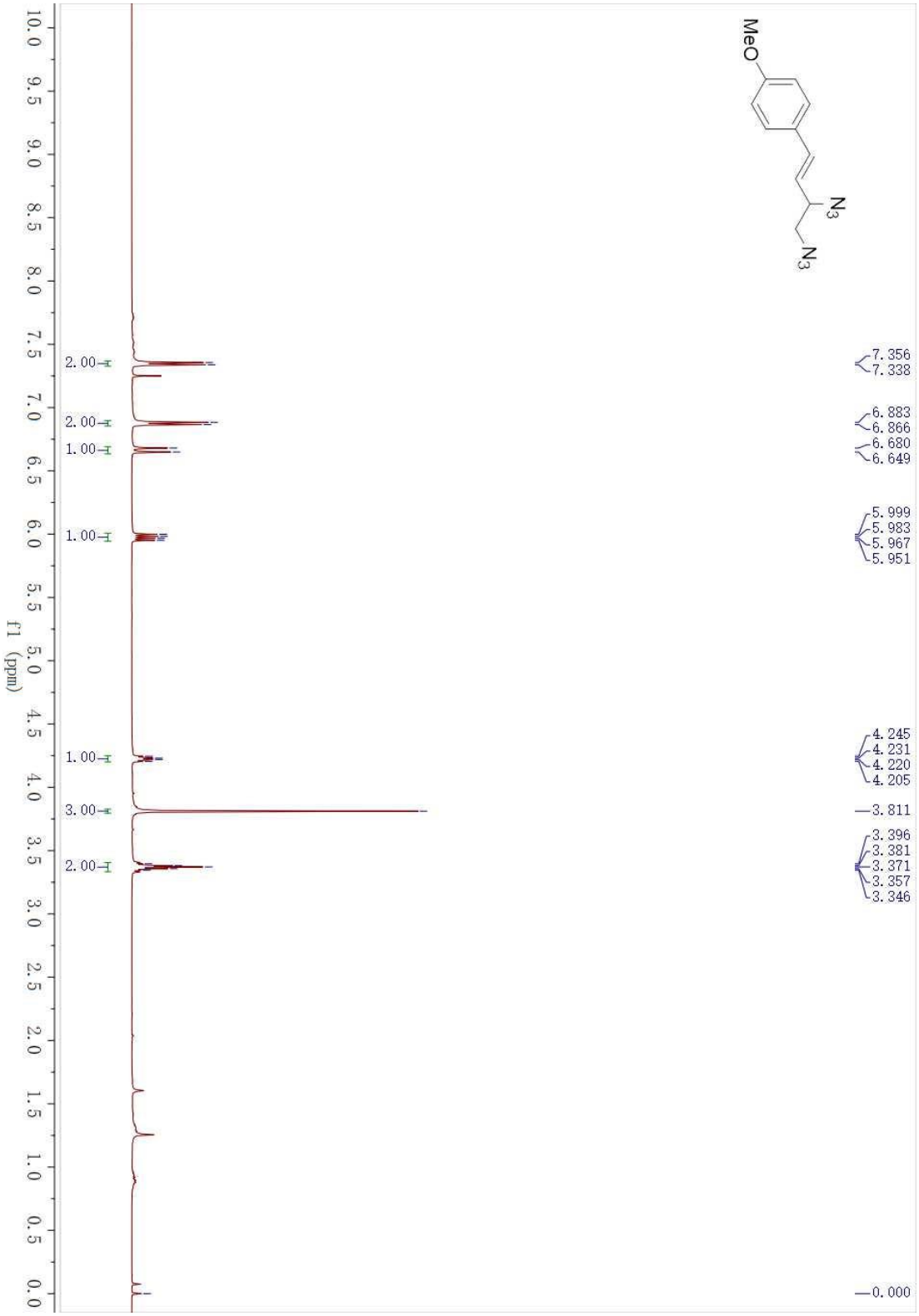

MeO
N3
CN
Br
160.020
135.259
134.826
134.073
133.524
132.539
132.457
129.235
128.914
128.139
128.083
127.918
122.685
122.468
122.233
121.899
119.528
114.148
77.255
77.000
76.746
62.469
61.732
55.321
40.947
40.075
33.930
33.355
f1 (ppm)

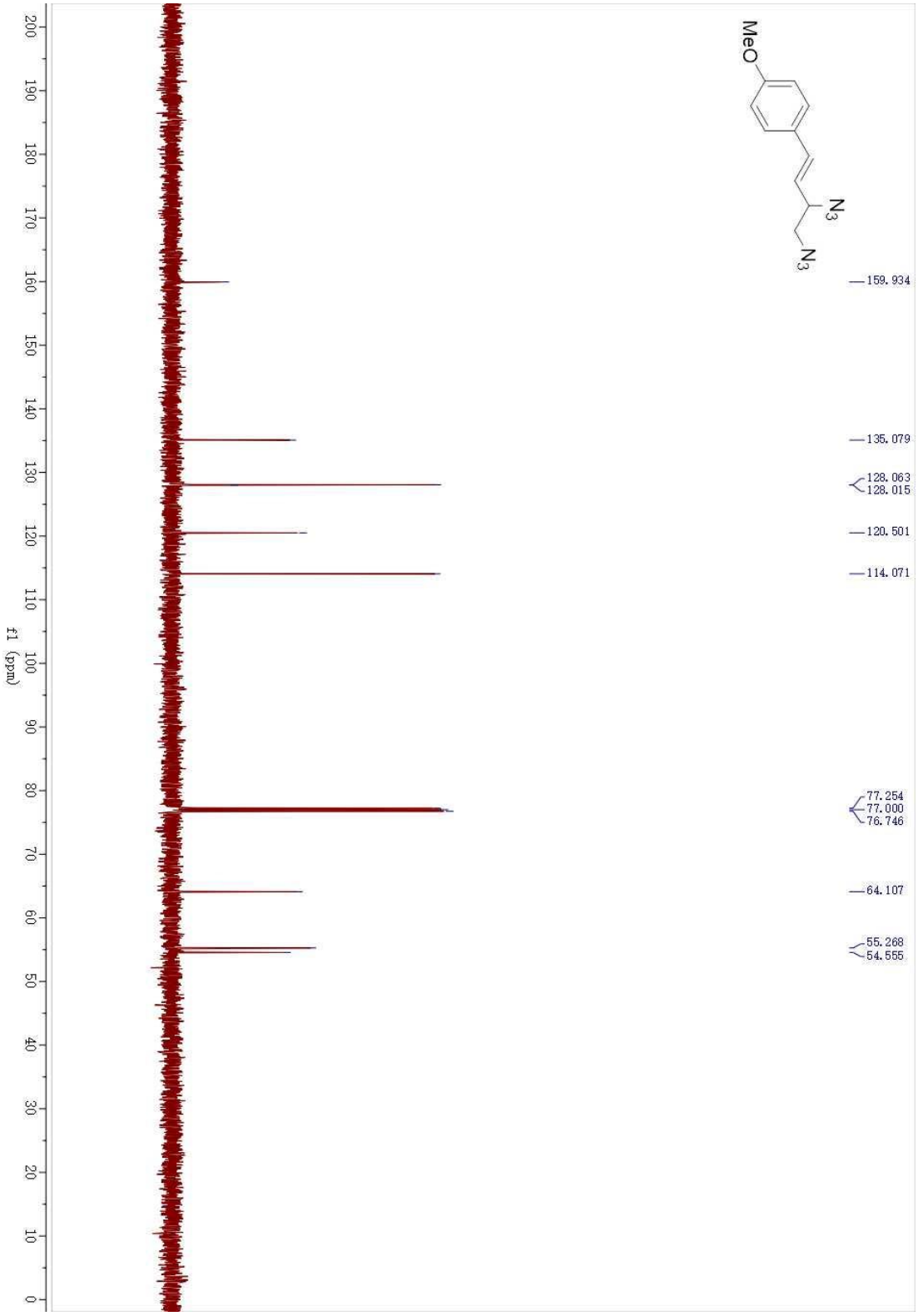
(*E*)-6-azido-2-(4-bromophenyl)-6-(p-tolyl)hex-4-enenitrile (5d):



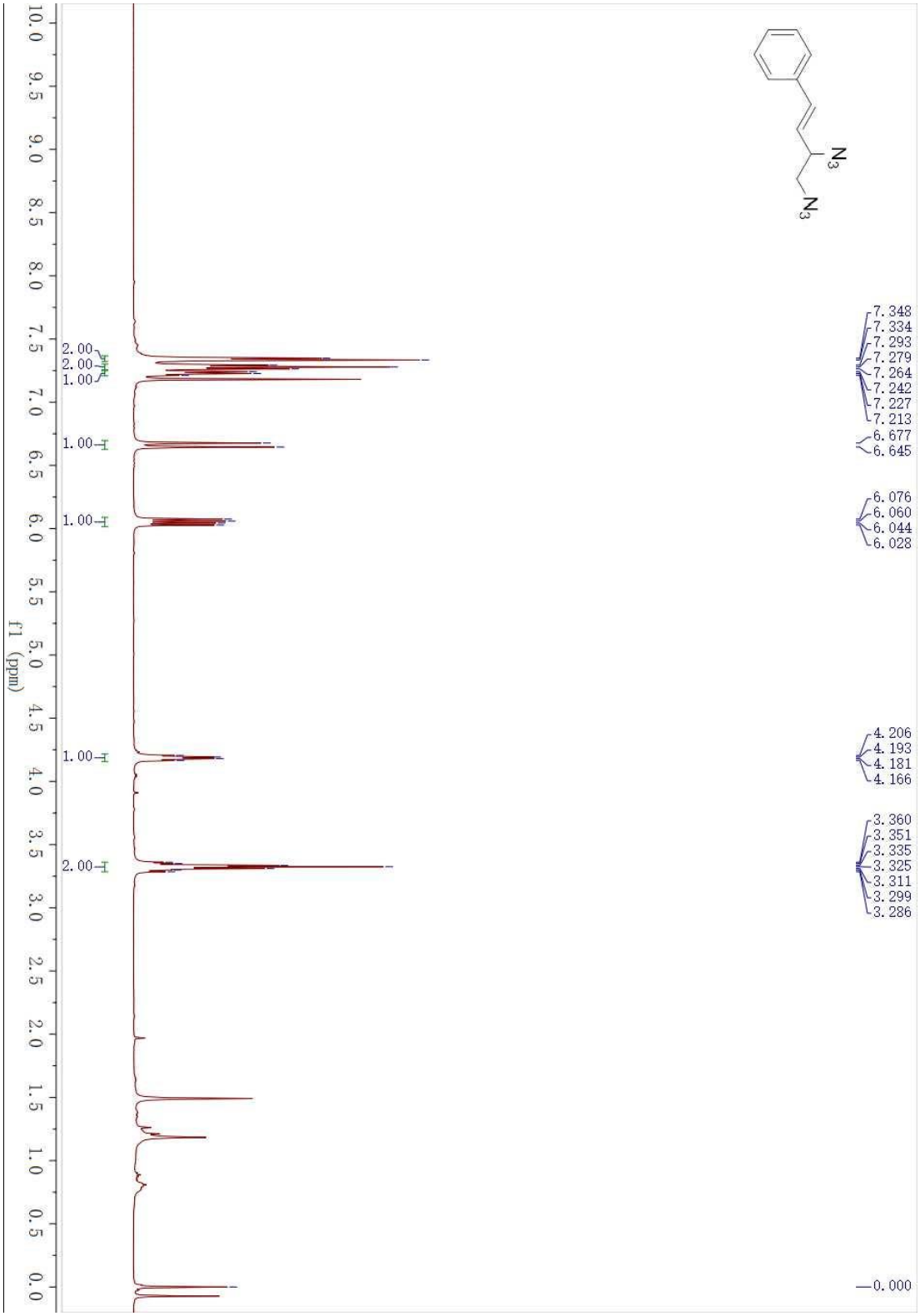

N3
CN
Br
138.763
138.679
135.574
135.151
134.024
133.462
132.502
132.417
129.410
129.204
128.883
126.716
126.669
123.473
123.169
122.654
122.434
119.821
119.479
77.254
77.000
76.747
62.306
61.566
40.816
39.960
33.872
33.298
21.216
f1 (ppm)

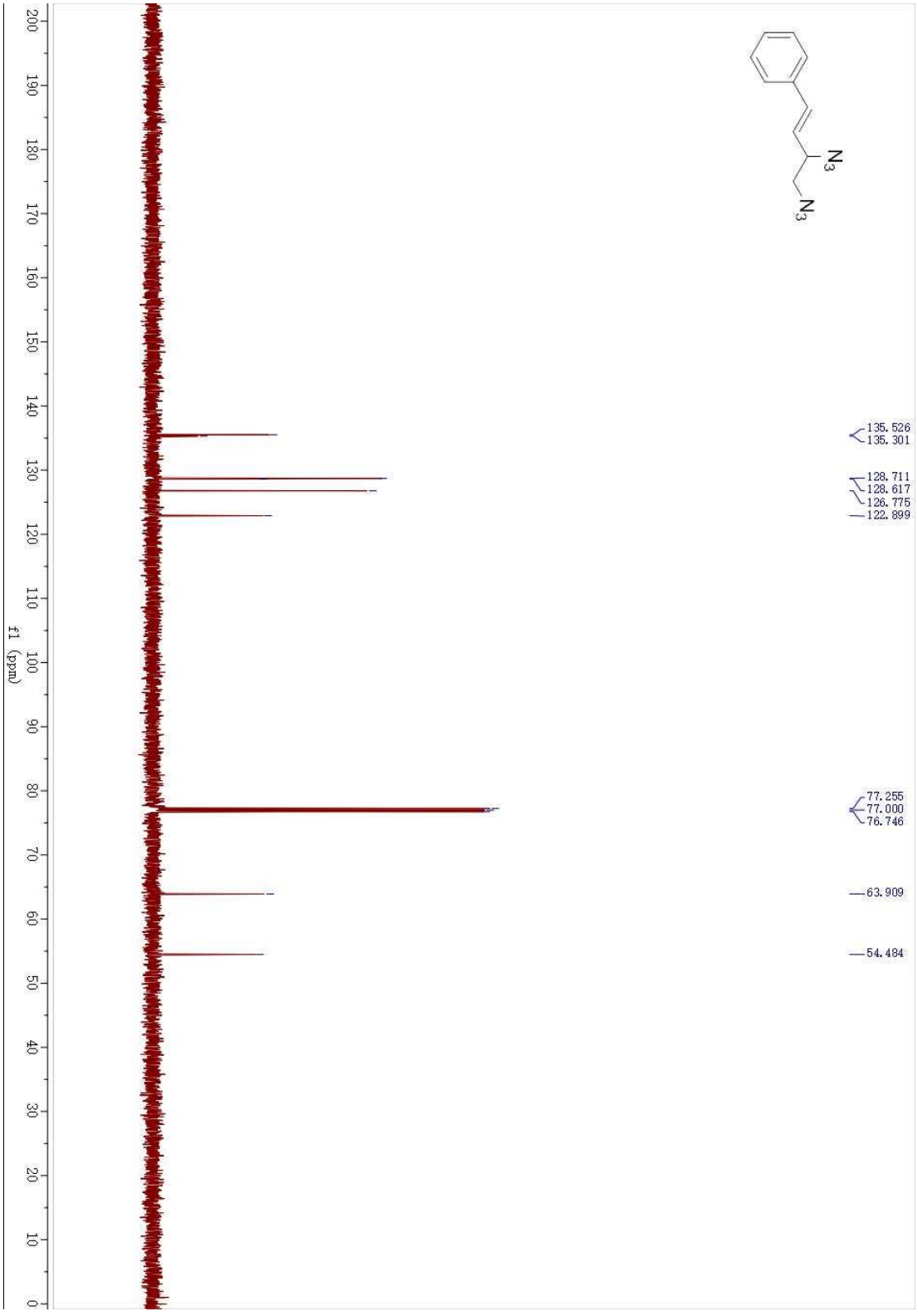
(*E*)-1-(3,4-diazidobut-1-en-1-yl)-4-methoxybenzene (4a):



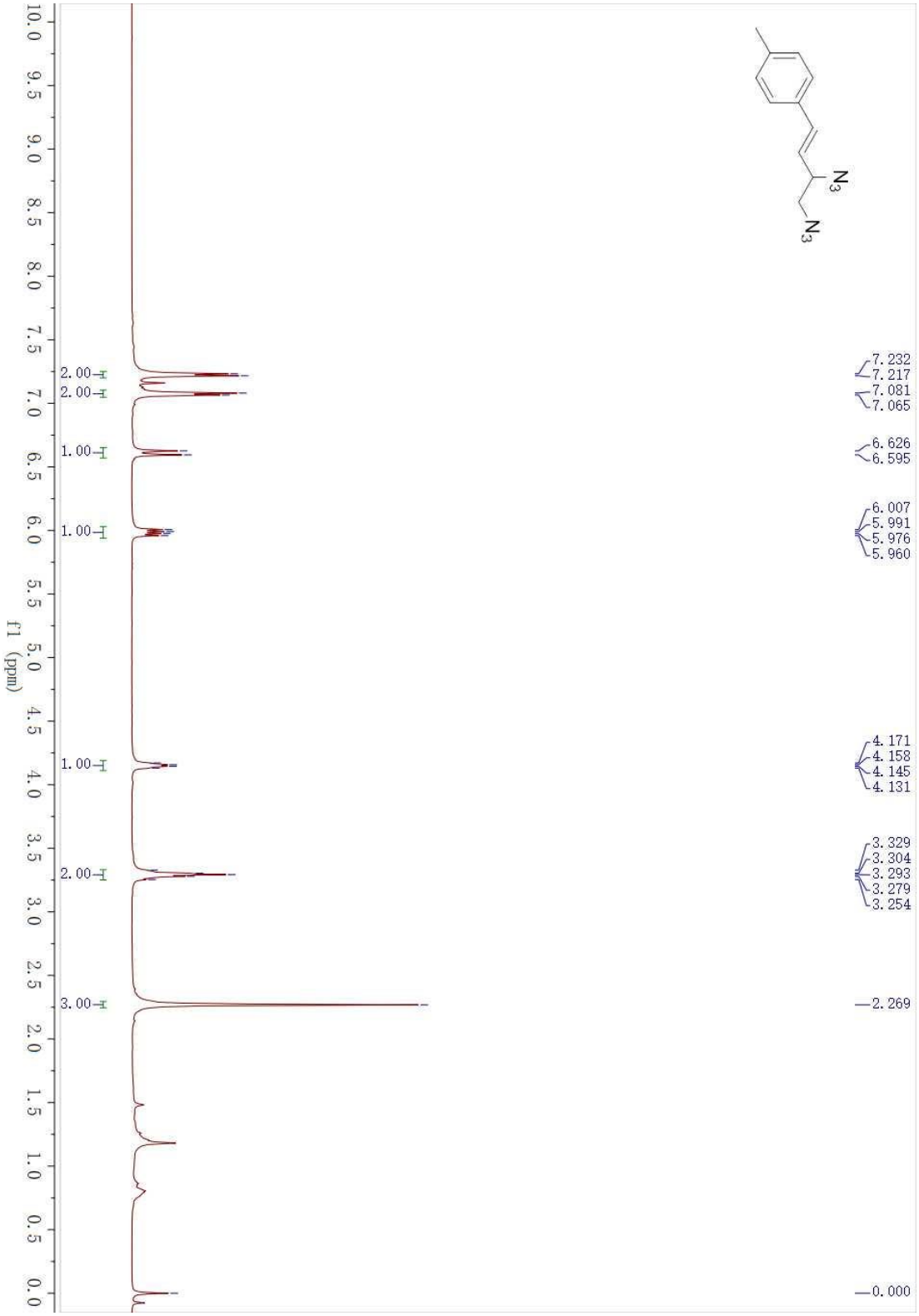

MeO
N3
N3
159.934
135.079
128.063
128.015
120.501
114.071
77.254
77.000
76.746
64.107
55.268
54.555
f1 (ppm)

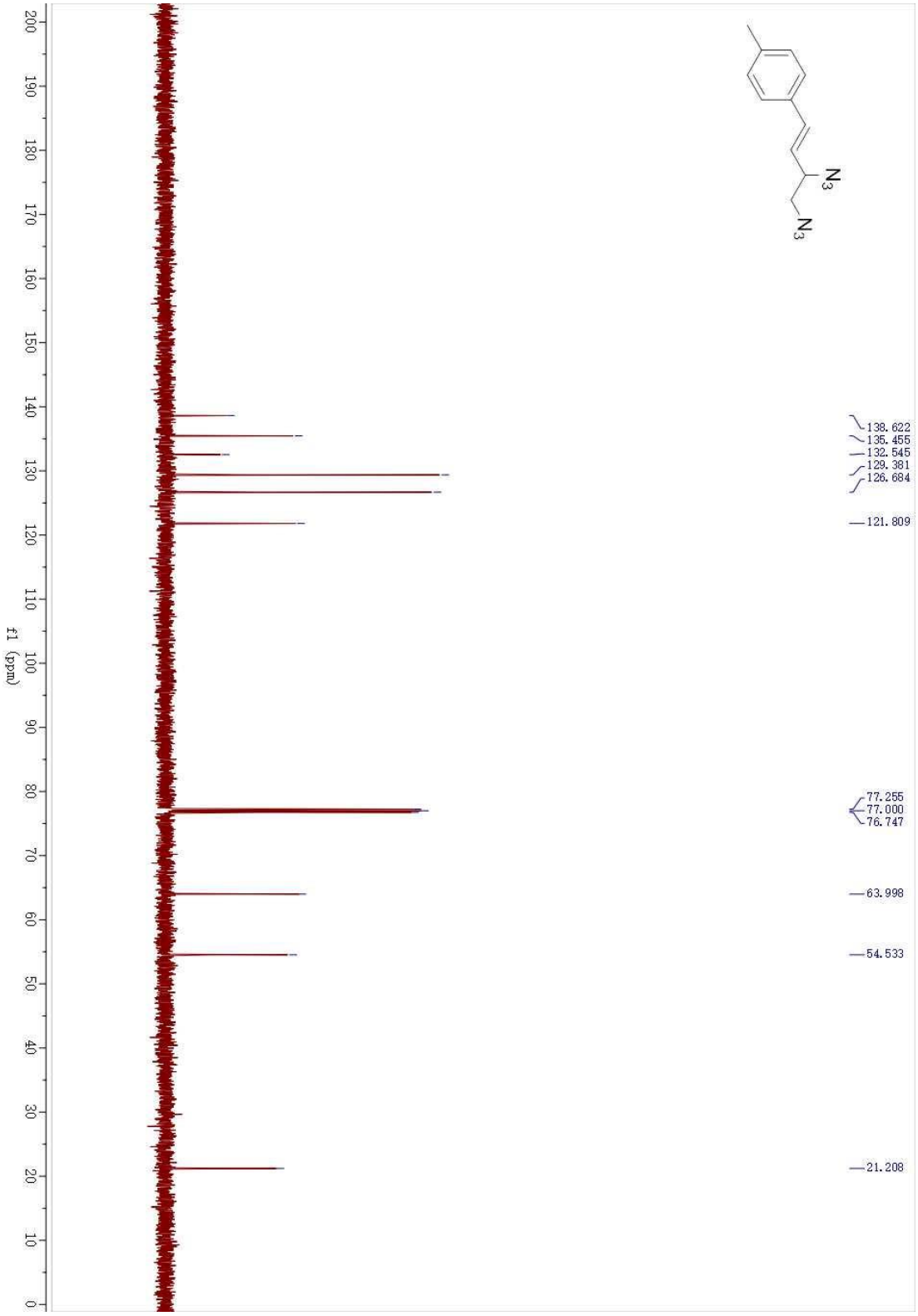
(*E*)-(3,4-diazidobut-1-en-1-yl)benzene (4b):



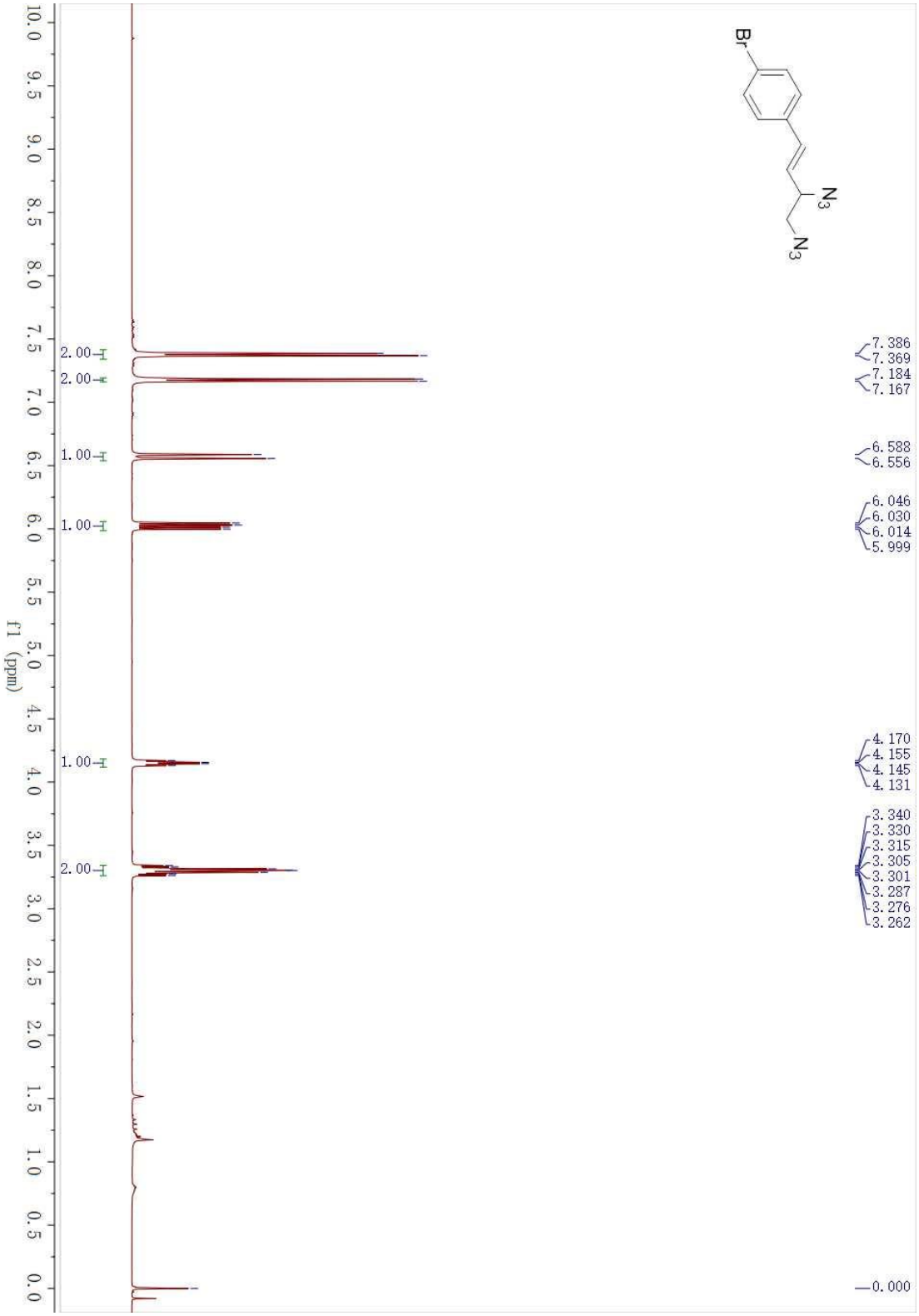

200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)
N3
N3
135.526
135.301
128.711
128.617
126.775
122.899
77.255
77.000
76.746
63.909
54.484

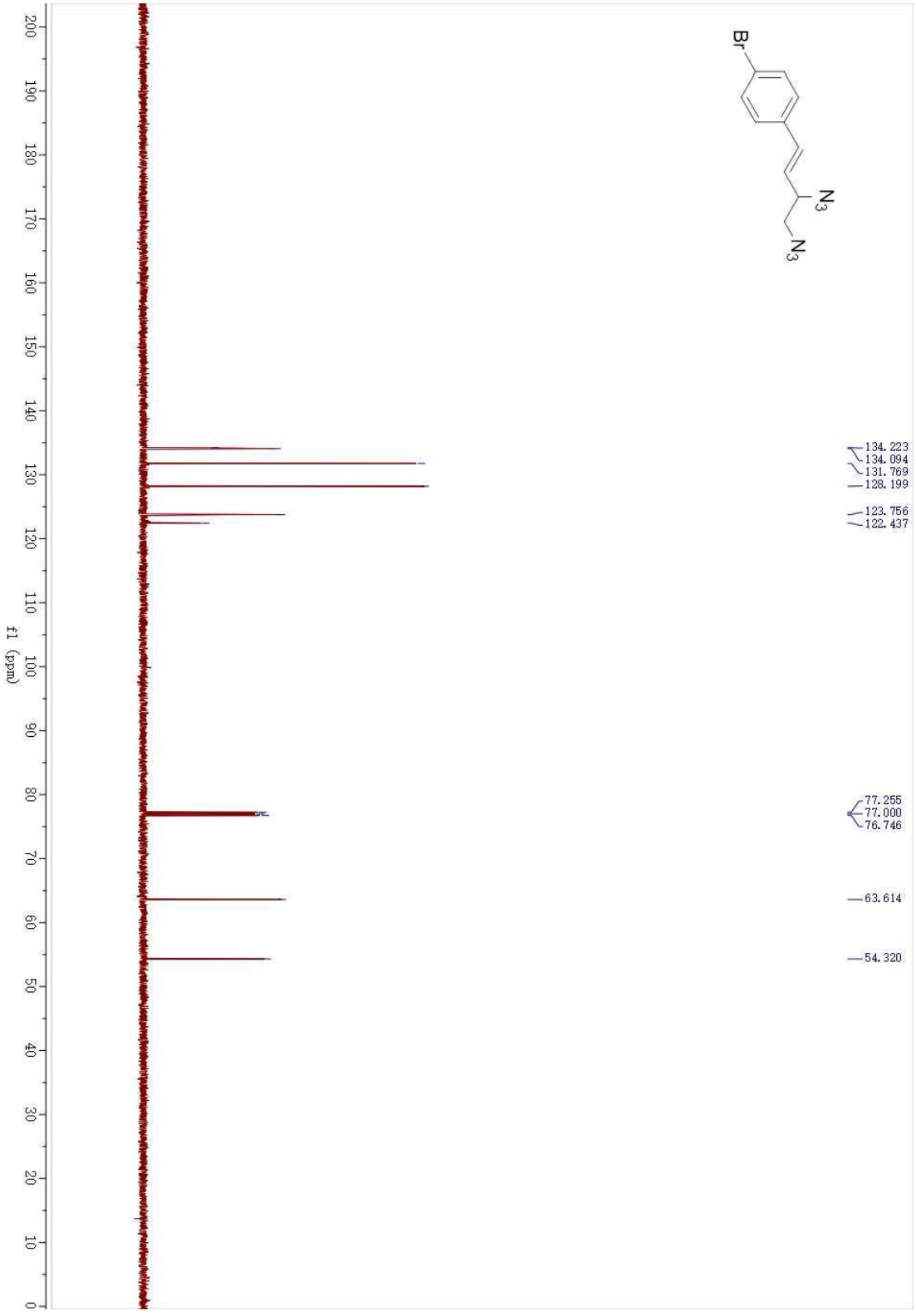
(*E*)-1-(3,4-diazidobut-1-en-1-yl)-4-methylbenzene (4c):



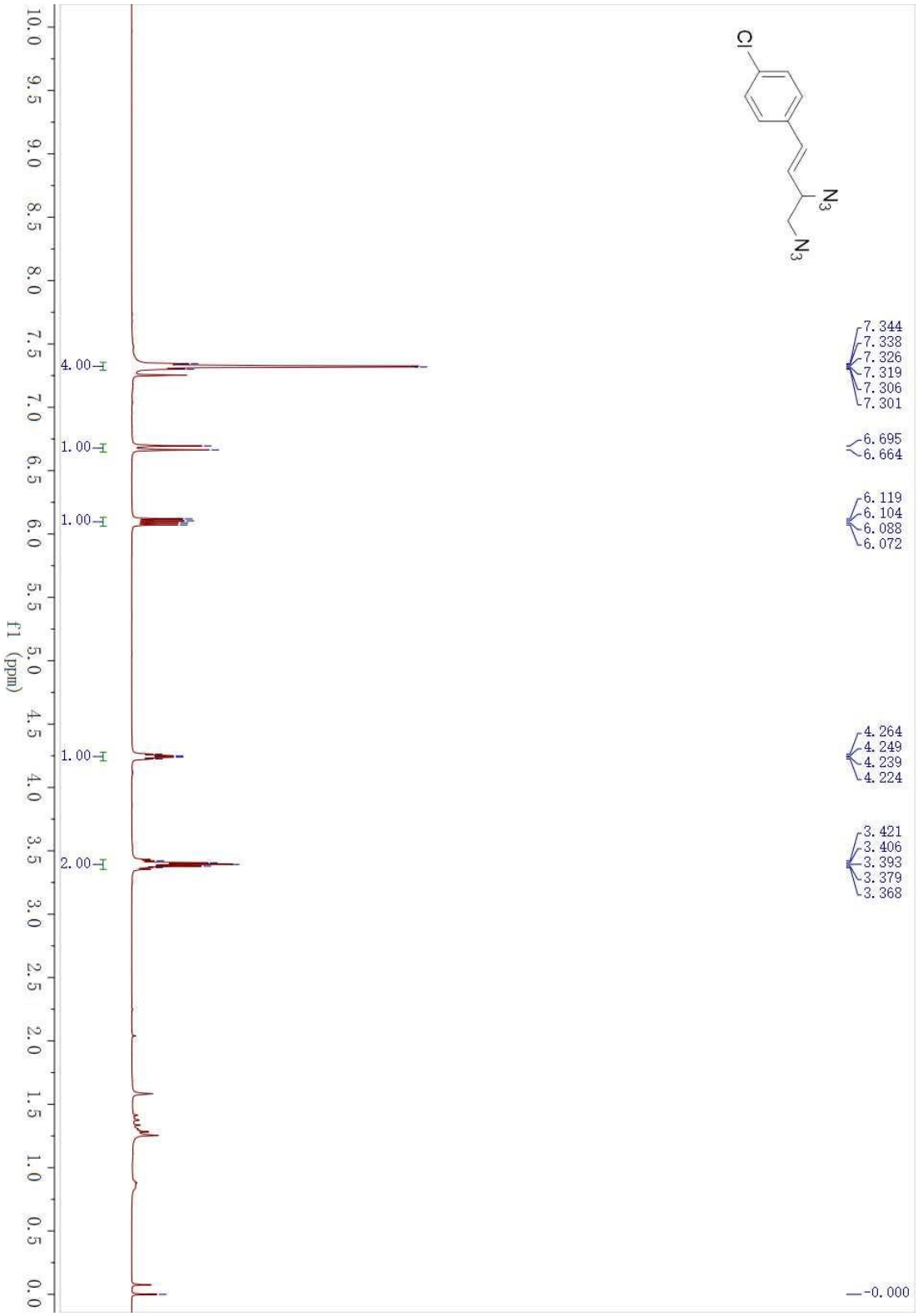

f1 (ppm)
138.622
135.455
132.545
129.381
126.684
121.809
77.255
77.000
76.747
63.998
54.533
21.208
N3
N3

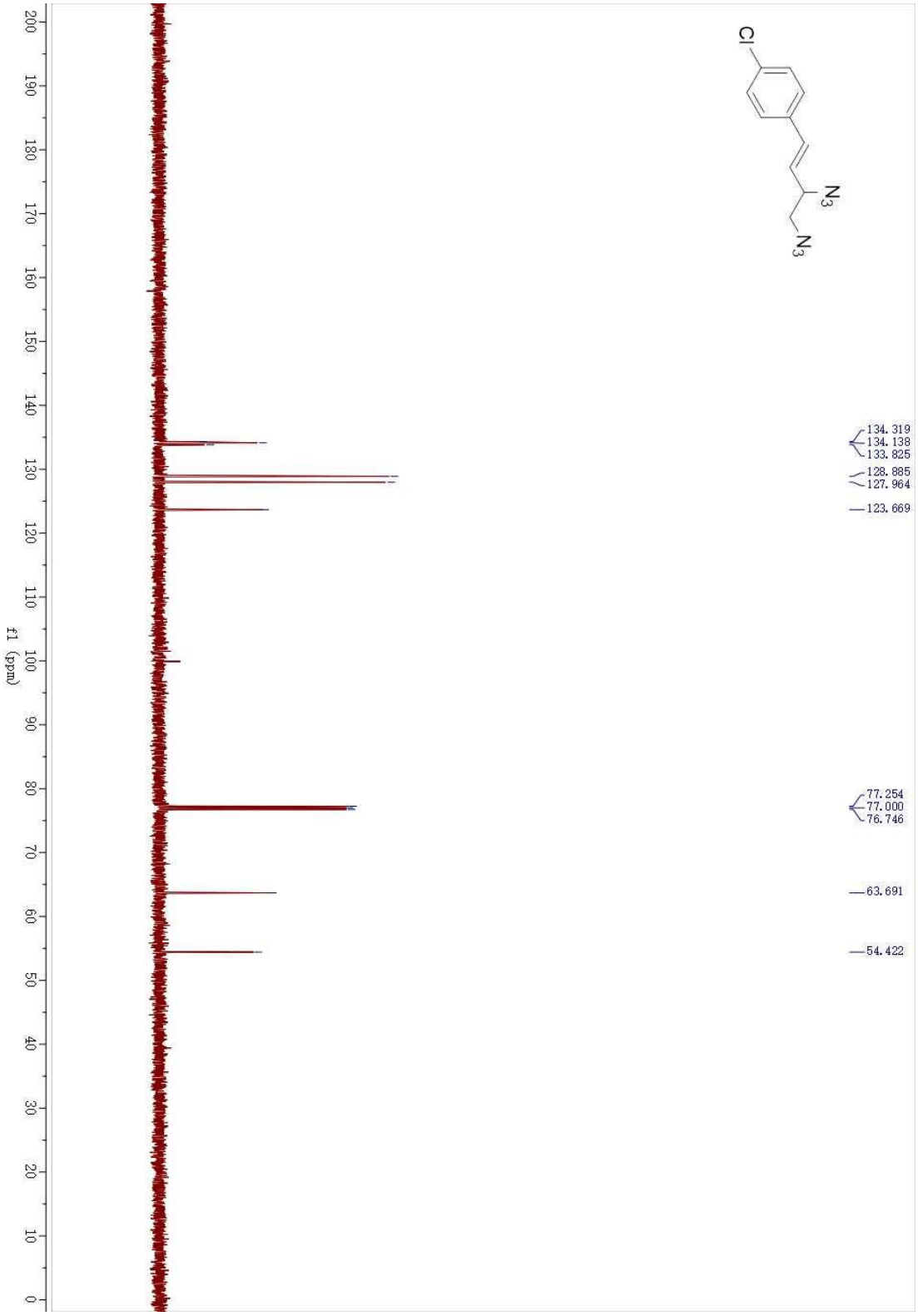
(*E*)-1-bromo-4-(3,4-diazidobut-1-en-1-yl)benzene (4d):



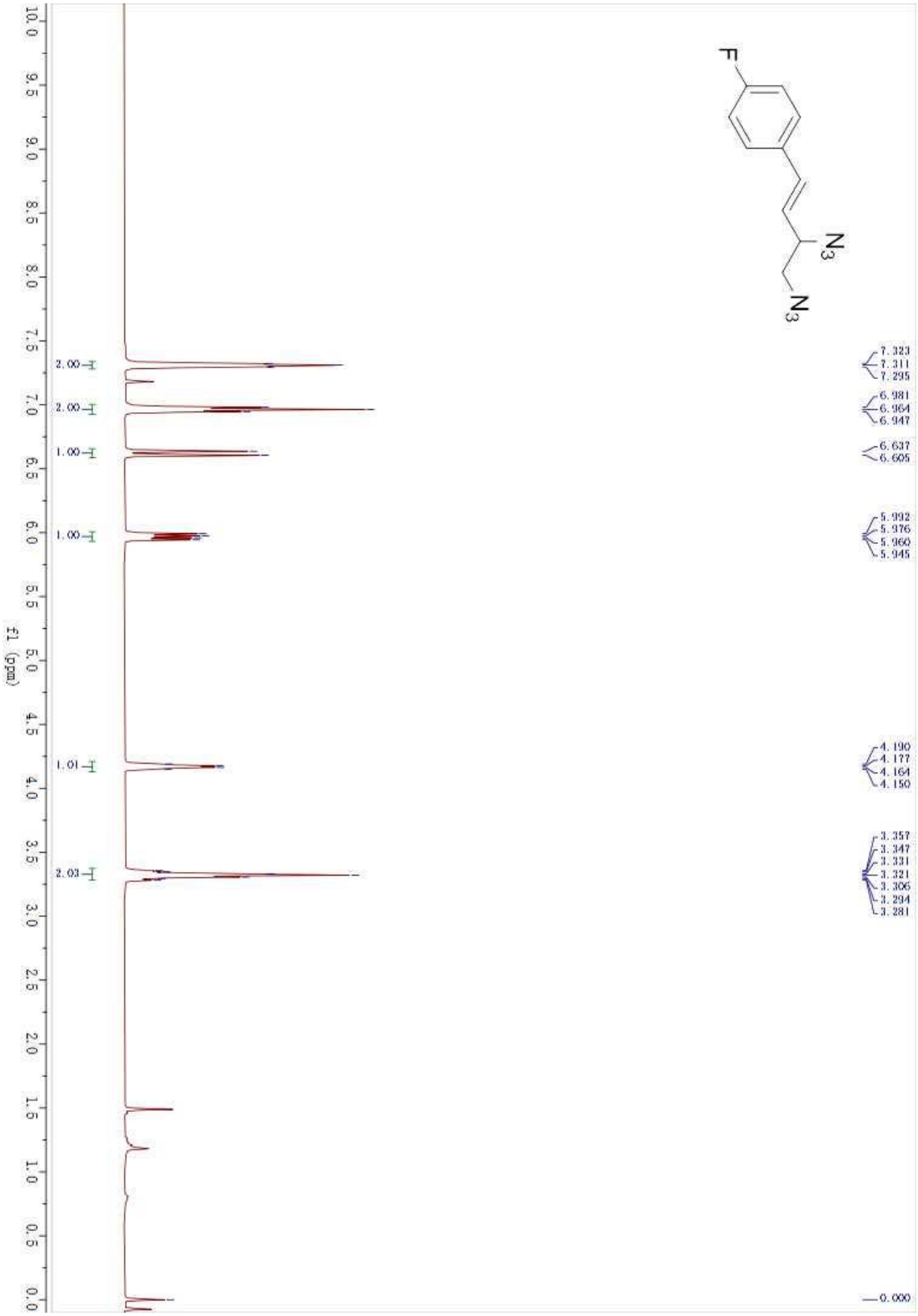

Br
N3
N3
134.223
134.094
131.769
128.199
123.756
122.437
77.255
77.000
76.746
63.614
54.320
f1 (ppm)

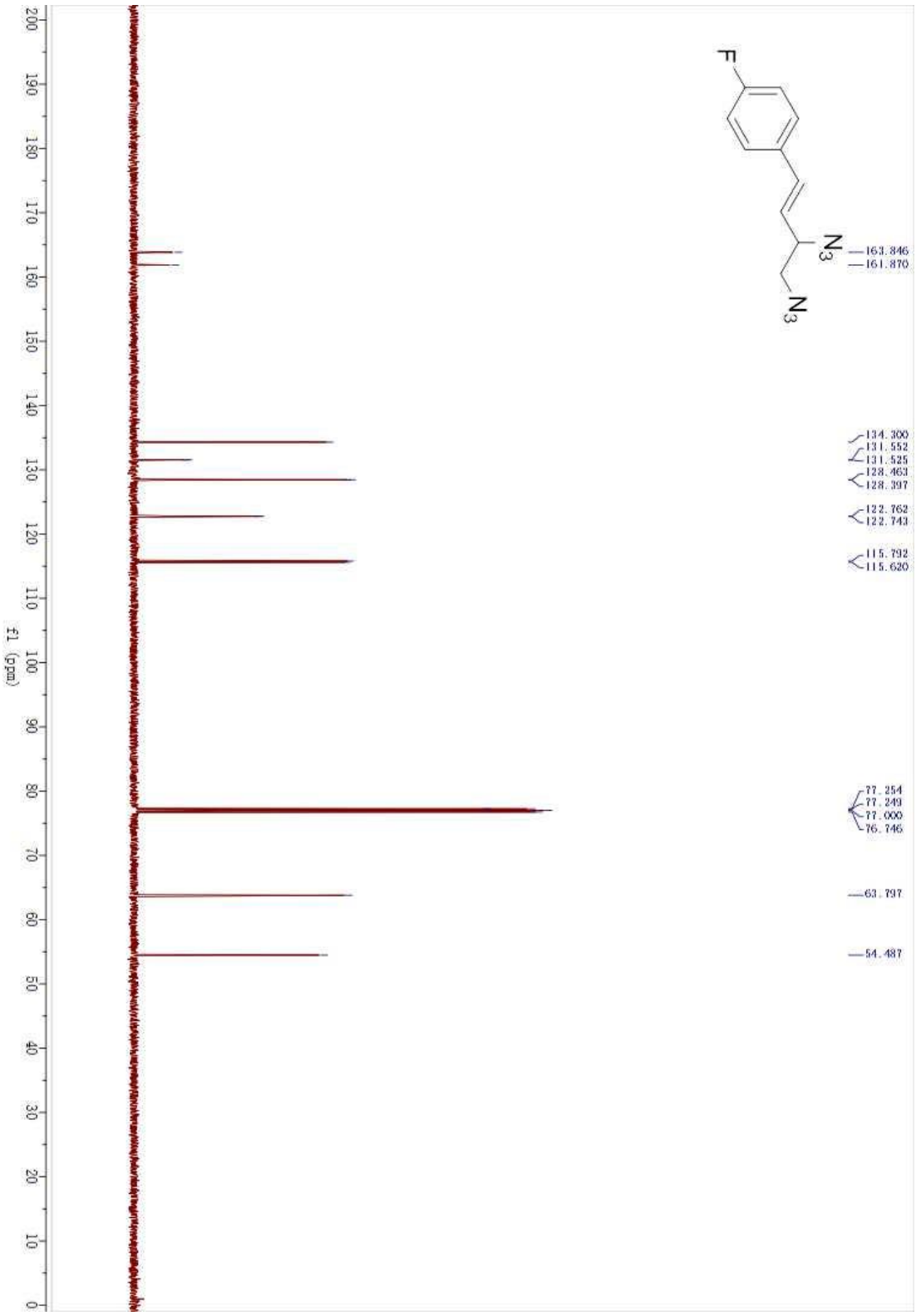
(*E*)-1-chloro-4-(3,4-diazidobut-1-en-1-yl)benzene (4e):

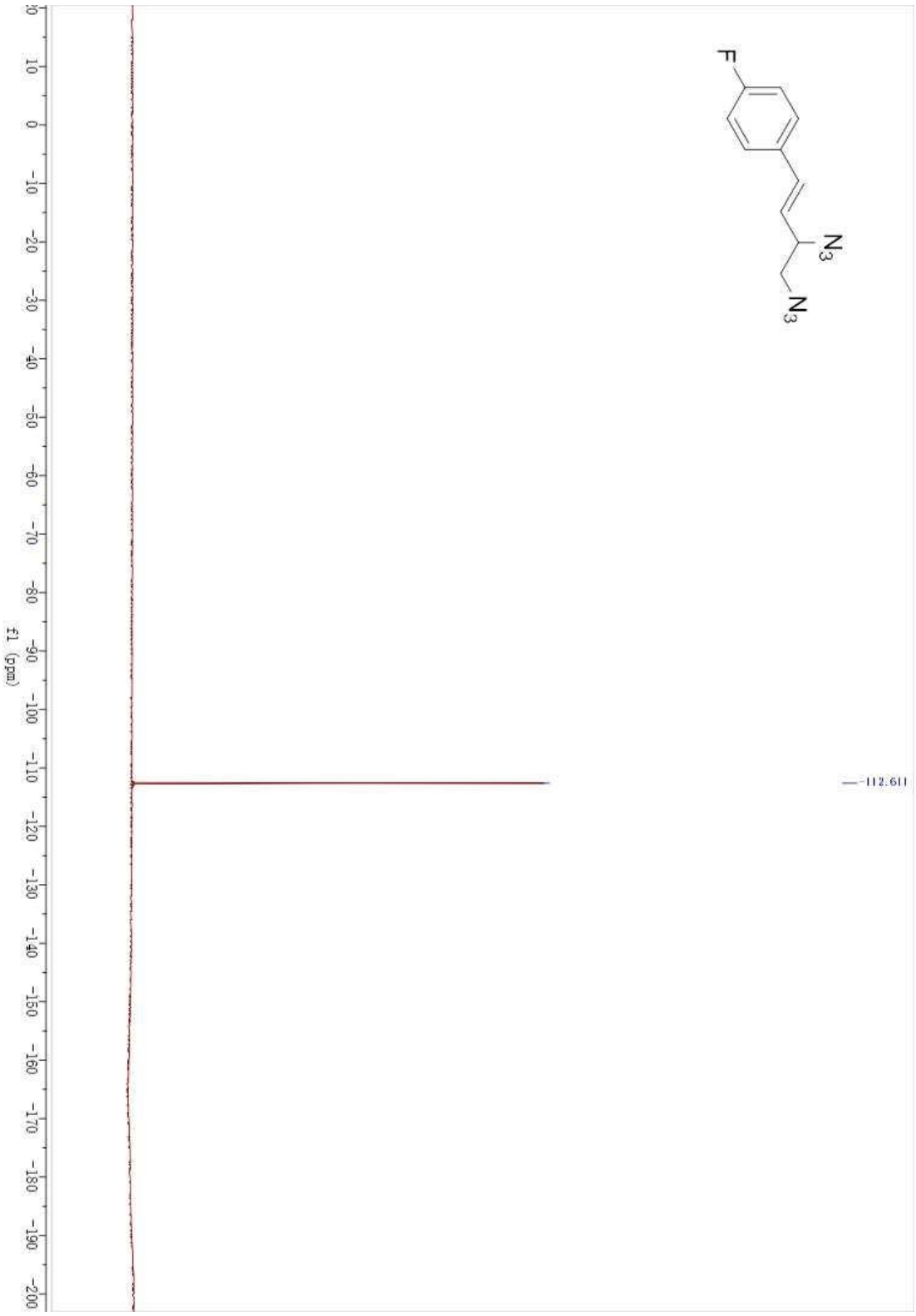



Cl
N3
N3
134.319
134.138
133.825
128.885
127.964
123.669
77.254
77.000
76.746
63.691
54.422
f1 (ppm)

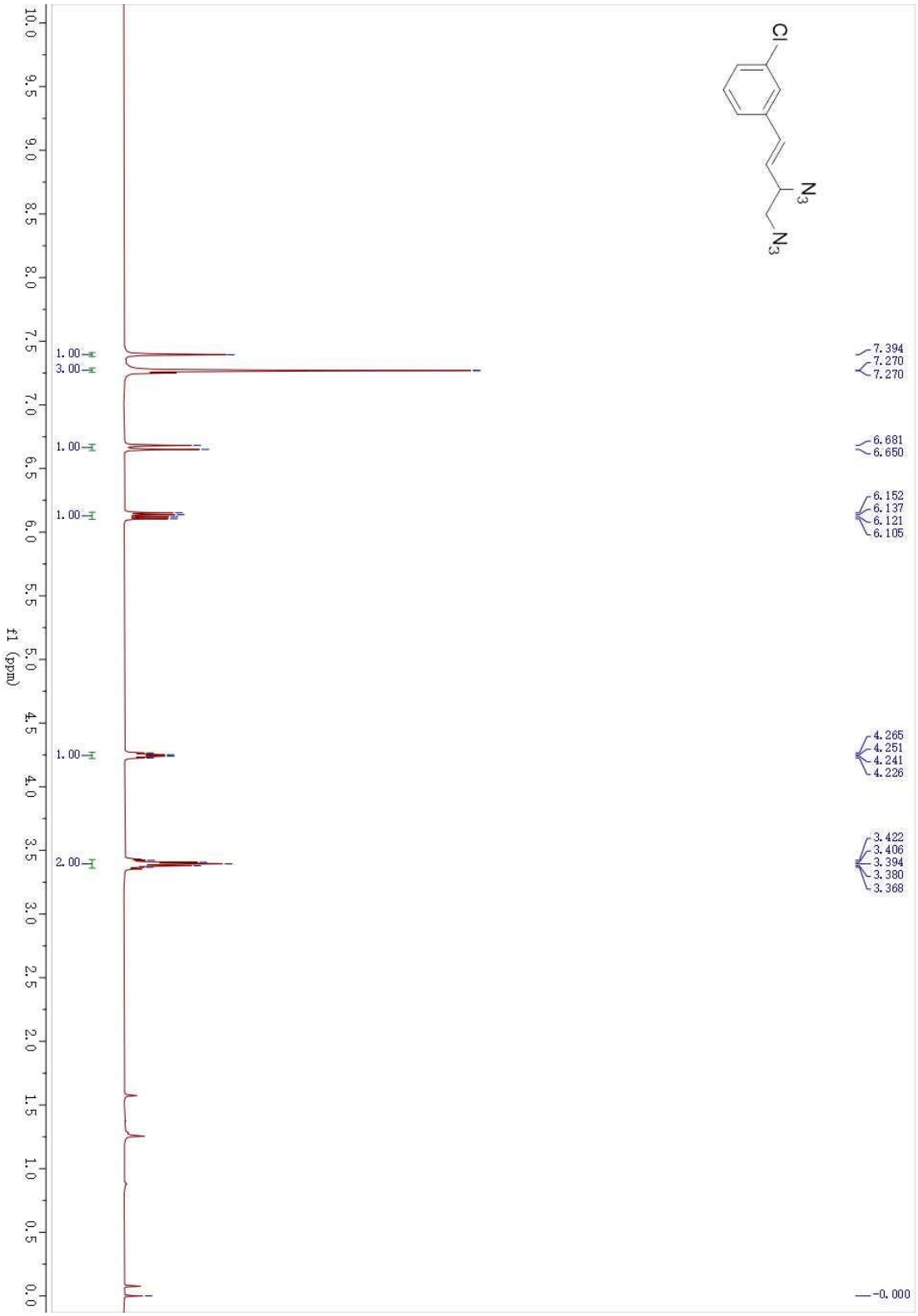
(*E*)-1-(3,4-diazidobut-1-en-1-yl)-4-fluorobenzene (4f):

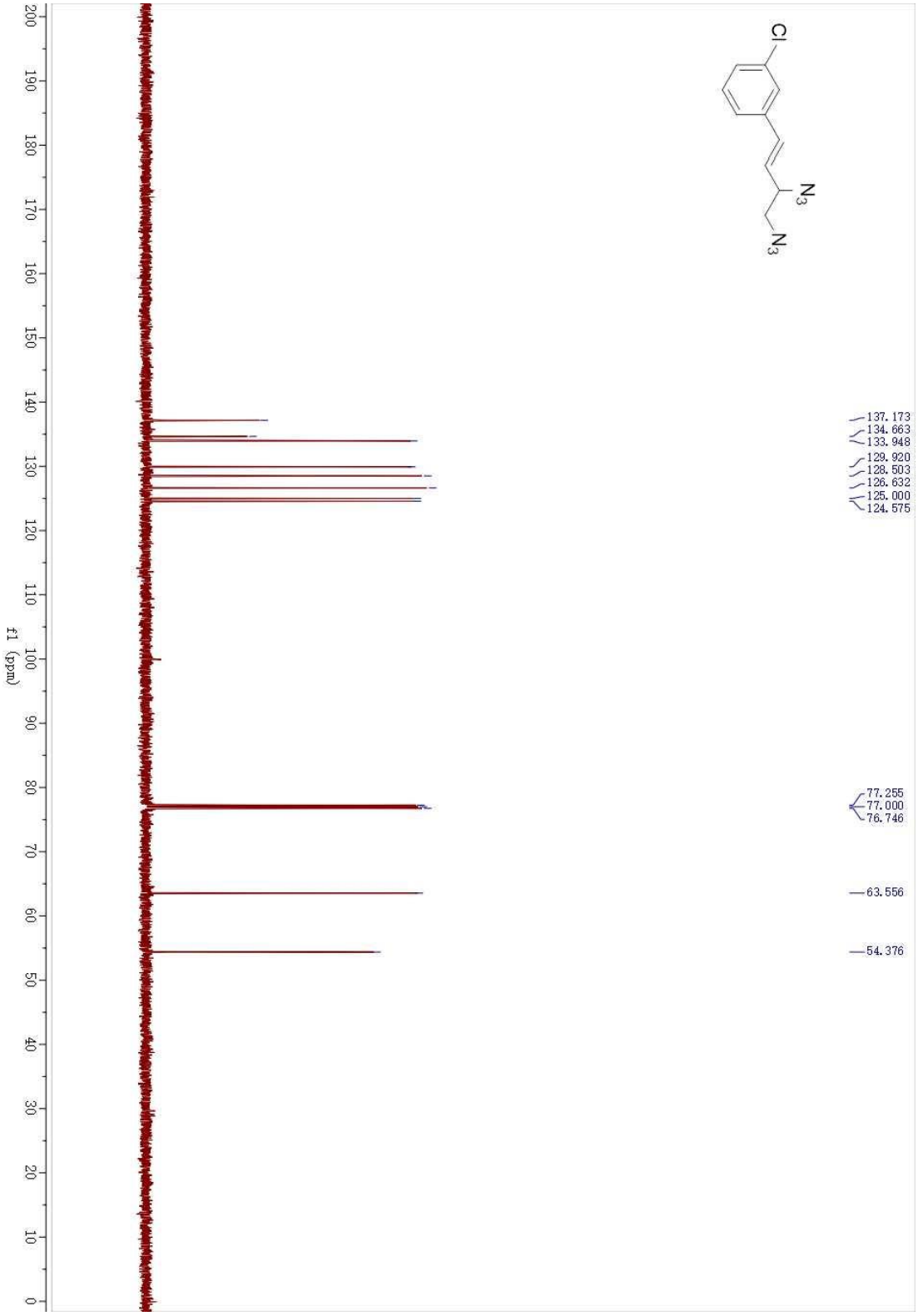



F
N3
N3
163.846
161.870
134.300
131.552
131.525
128.463
128.397
122.762
122.743
115.792
115.620
77.254
77.249
77.000
76.746
63.797
54.487
f1 (ppm)

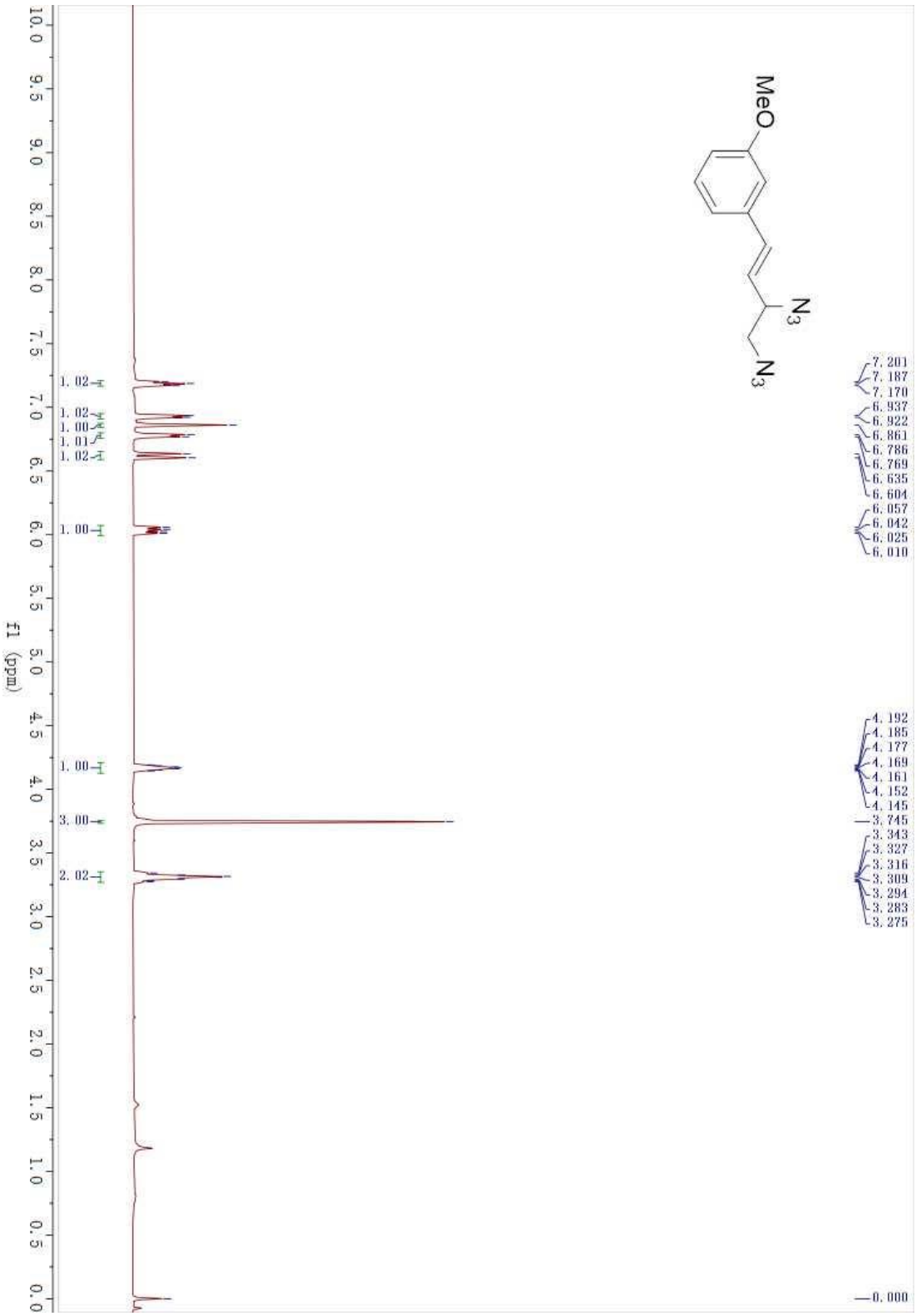

F
N3
N3
-112.611
f1 (ppm)
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200

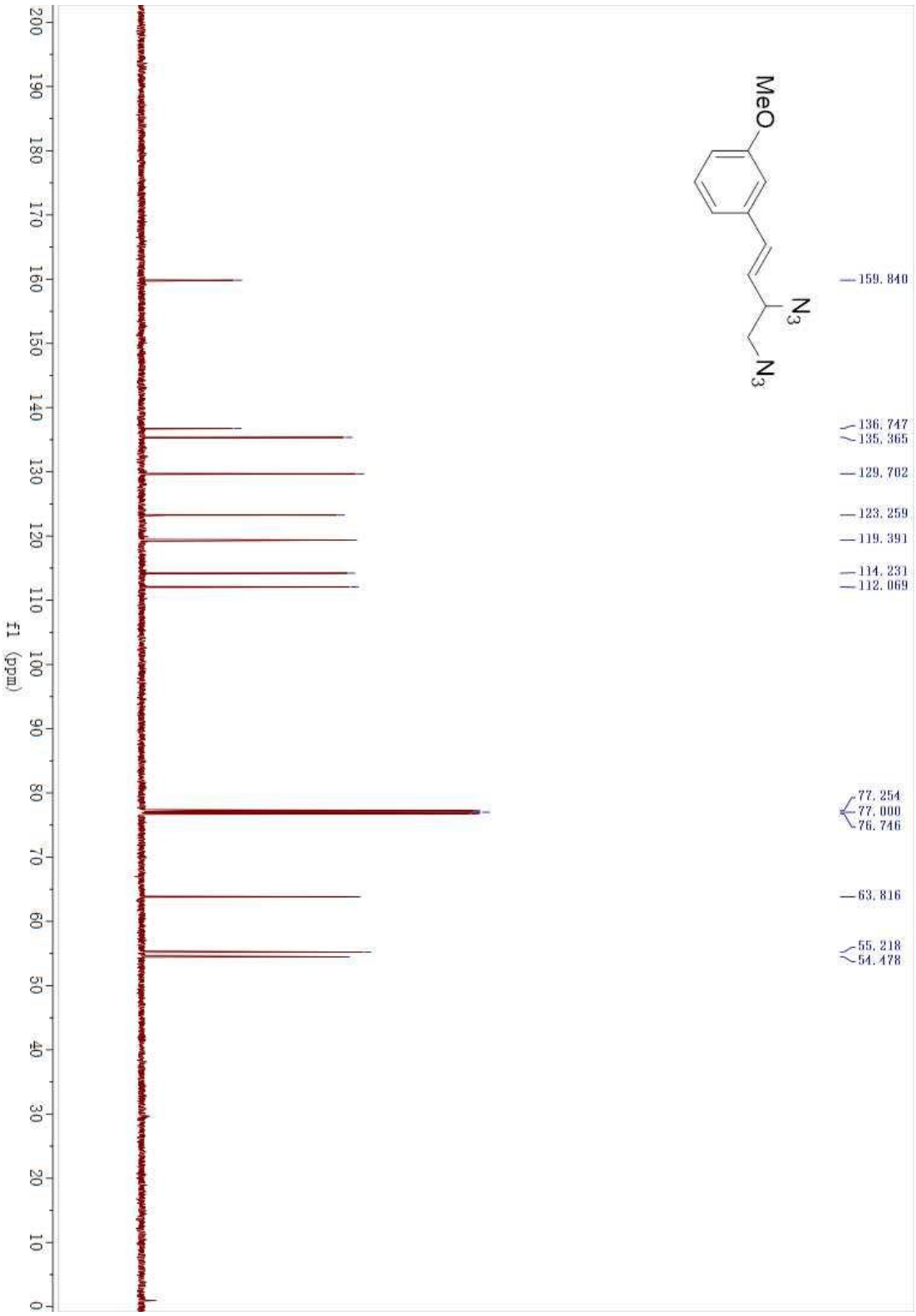
(*E*)-1-chloro-3-(3,4-diazidobut-1-en-1-yl)benzene (4g):



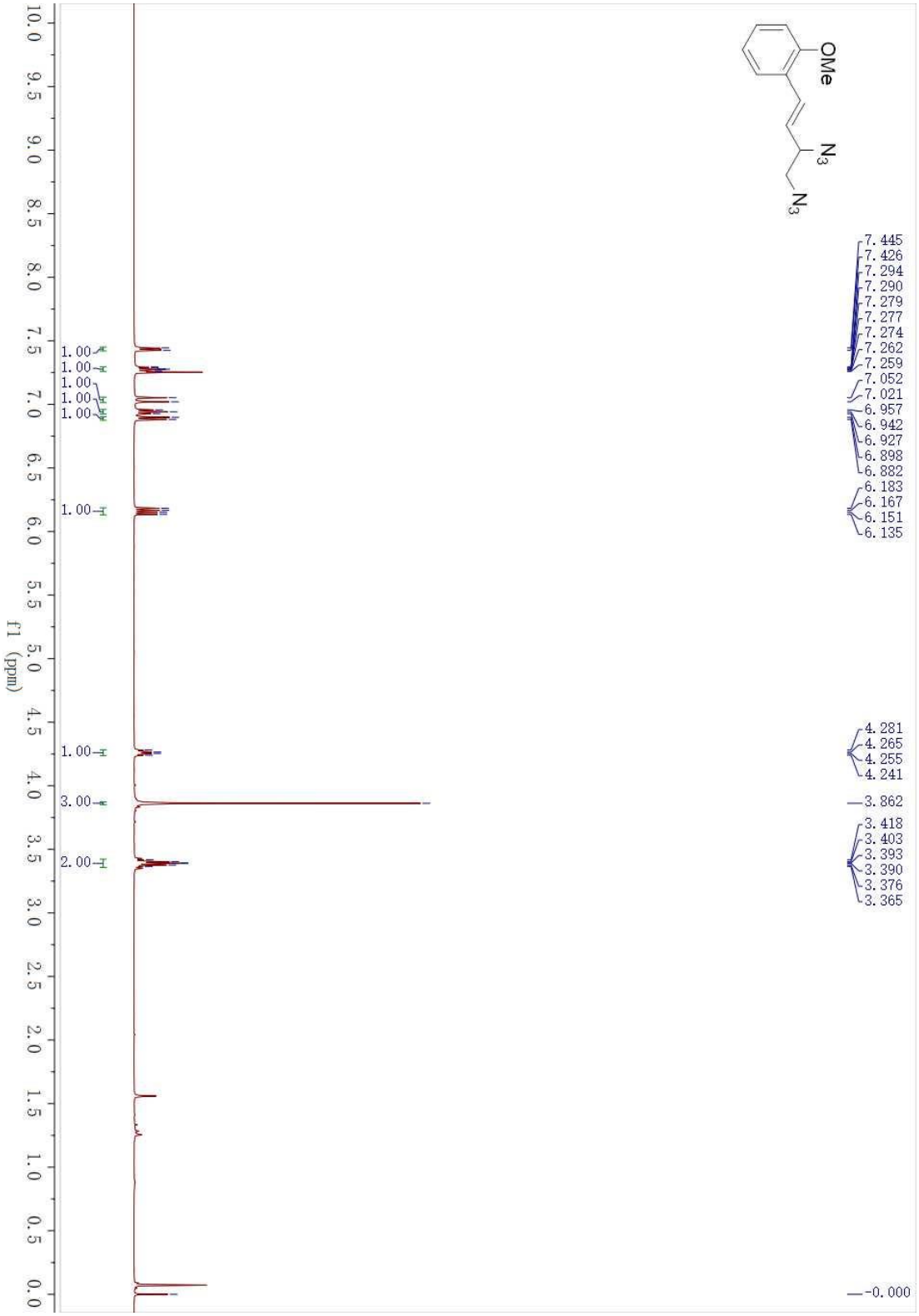

Cl
N3
N3
137.173
134.663
133.948
129.920
128.503
126.632
125.000
124.575
77.255
77.000
76.746
63.556
54.376
f1 (ppm)

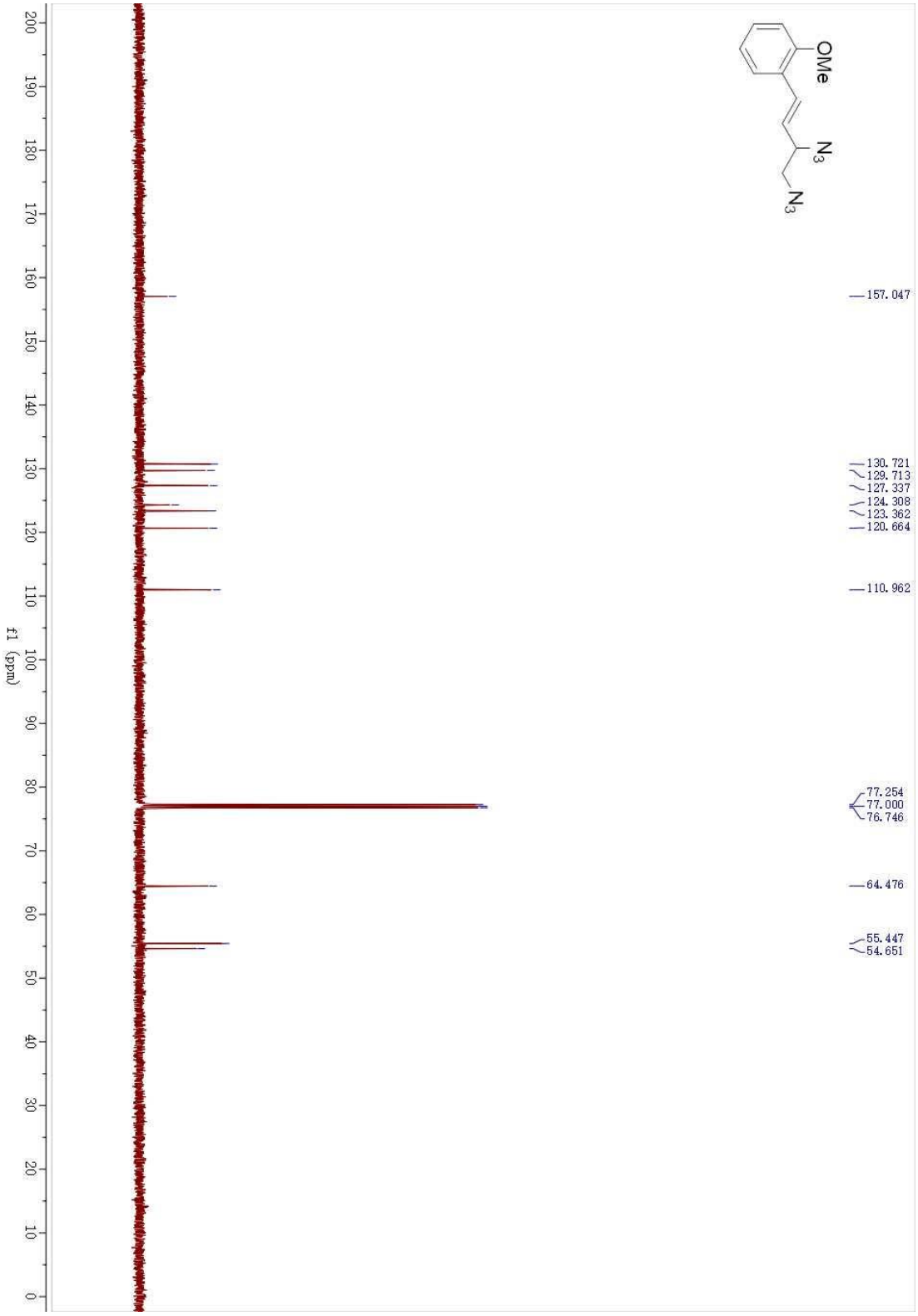
(E)-1-(3,4-diazidobut-1-en-1-yl)-3-methoxybenzene (4h):



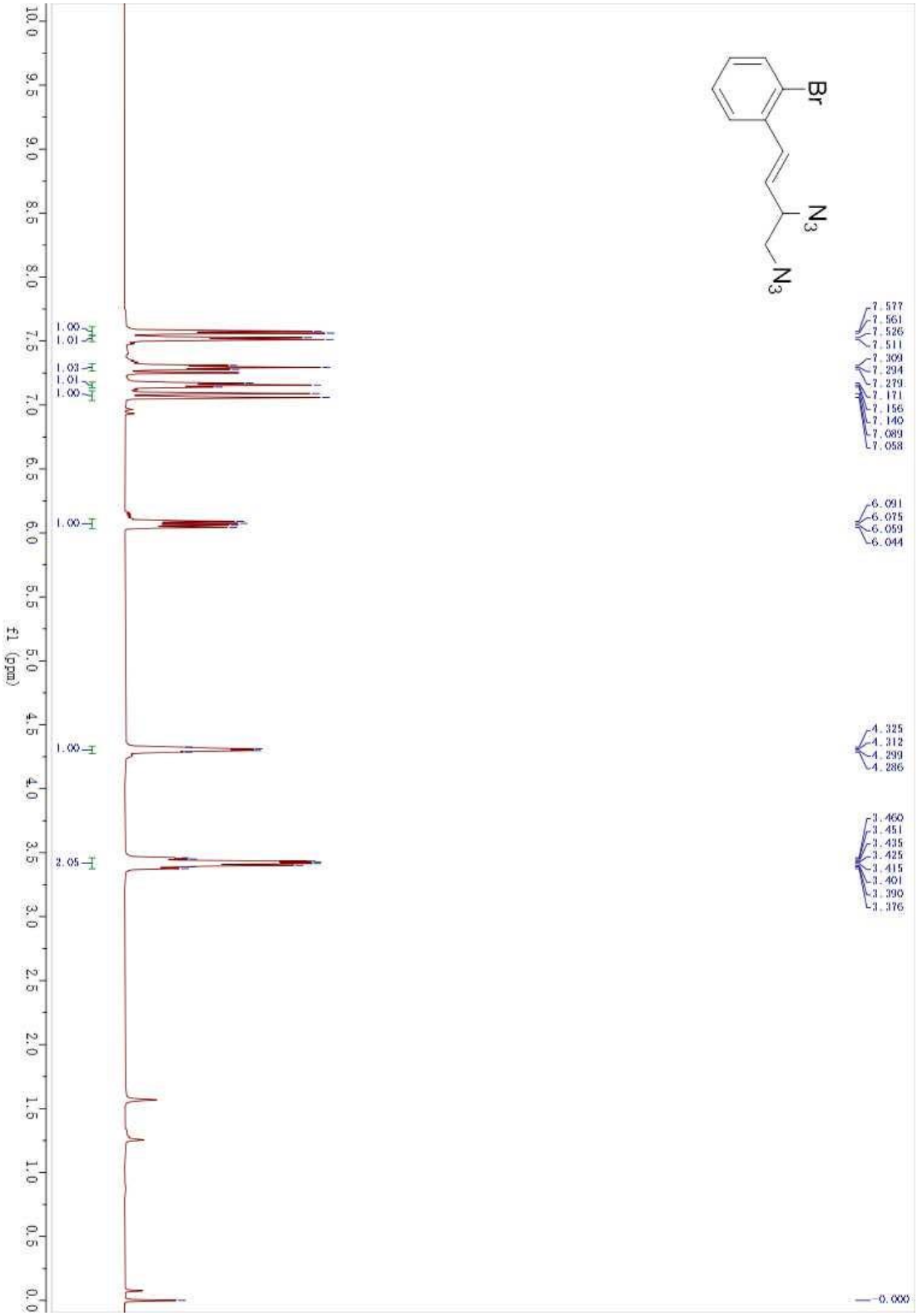

MeO
N3
N3
159.840
136.747
135.365
129.702
123.259
119.391
114.231
112.069
77.254
77.000
76.746
63.816
55.218
54.478
f1 (ppm)

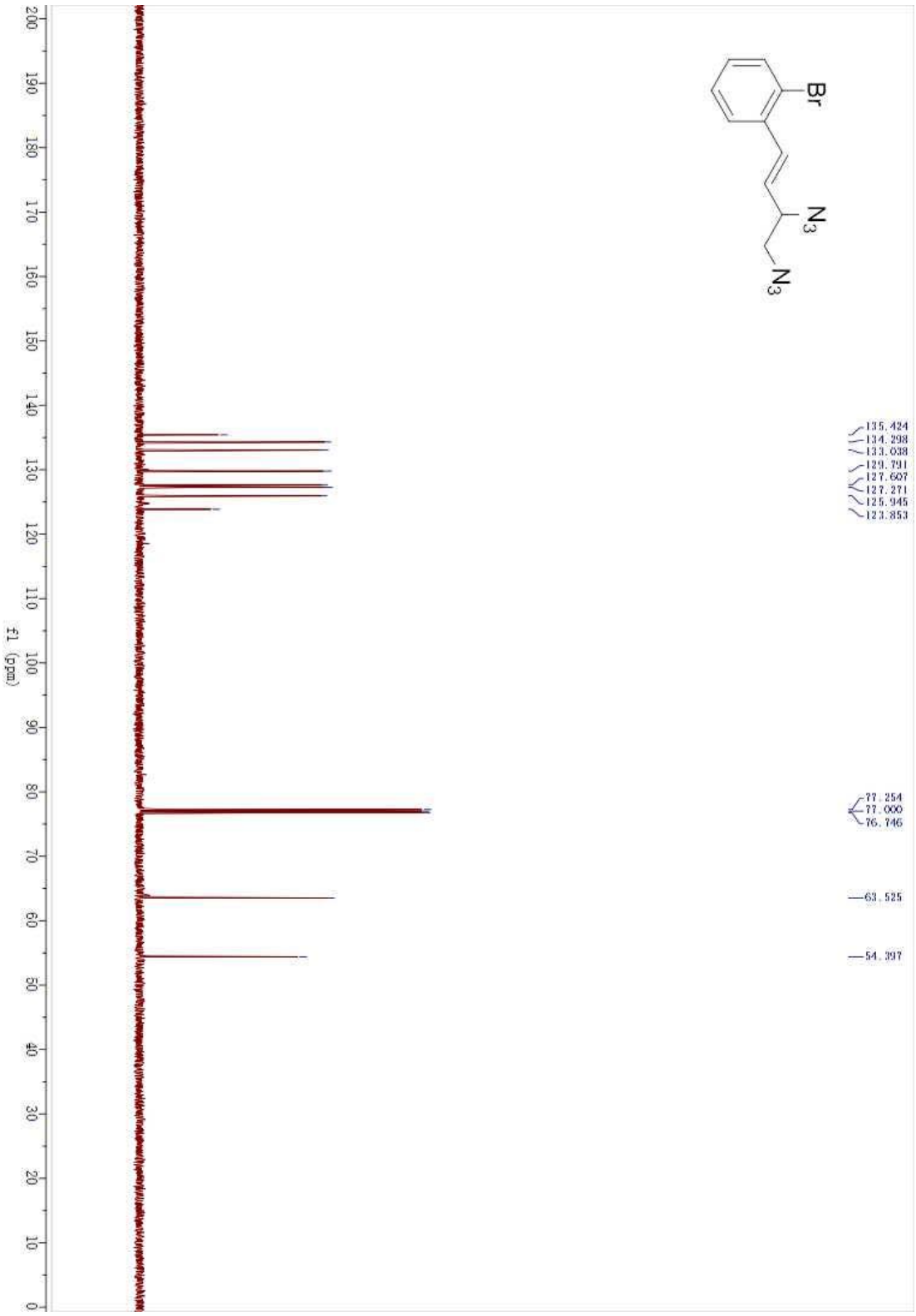
(*E*)-1-(3,4-diazidobut-1-en-1-yl)-2-methoxybenzene (4i):



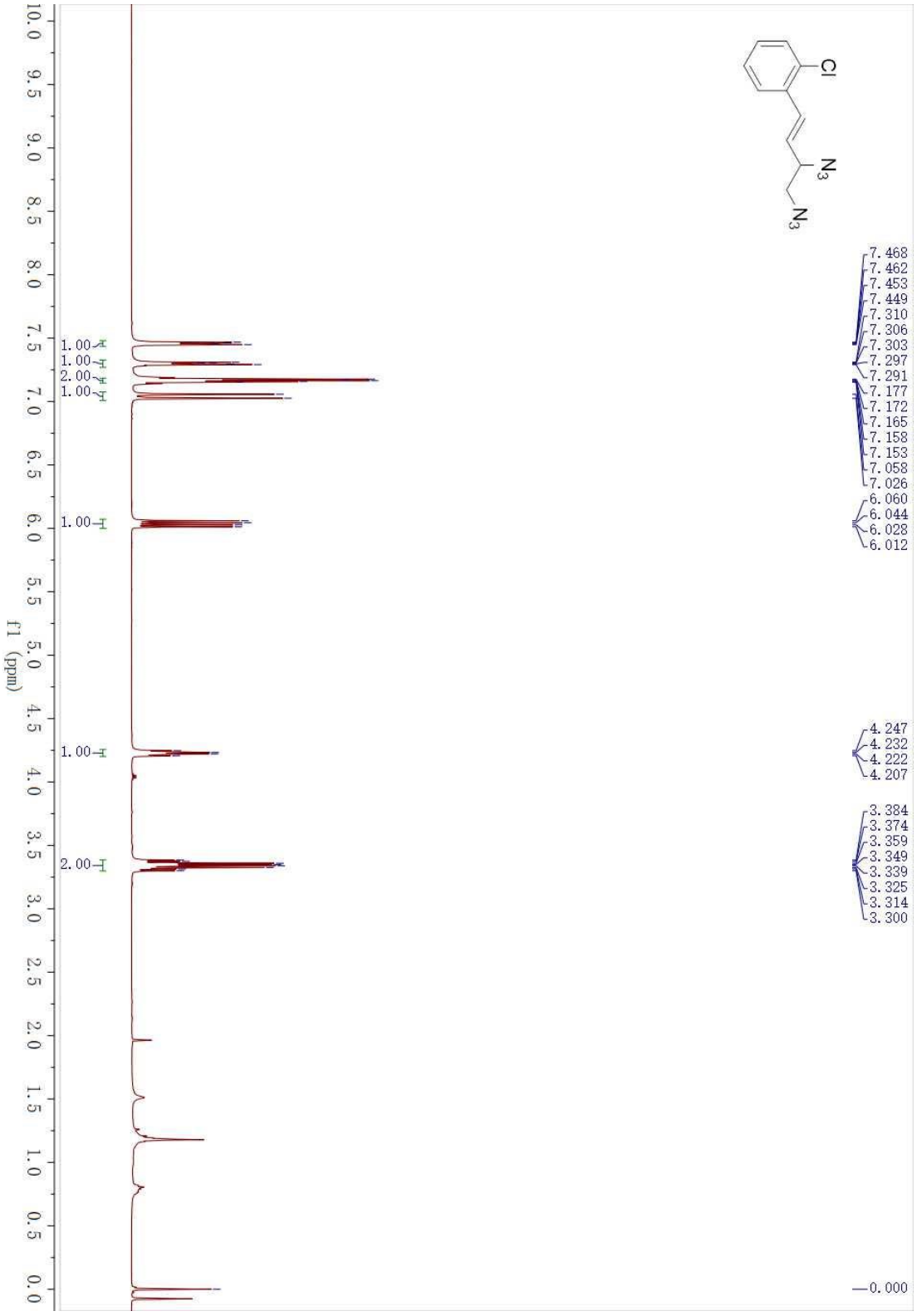

OMe
N3
N3
157.047
130.721
129.713
127.337
124.308
123.362
120.664
110.962
77.254
77.000
76.746
64.476
55.447
54.651
f1 (ppm)

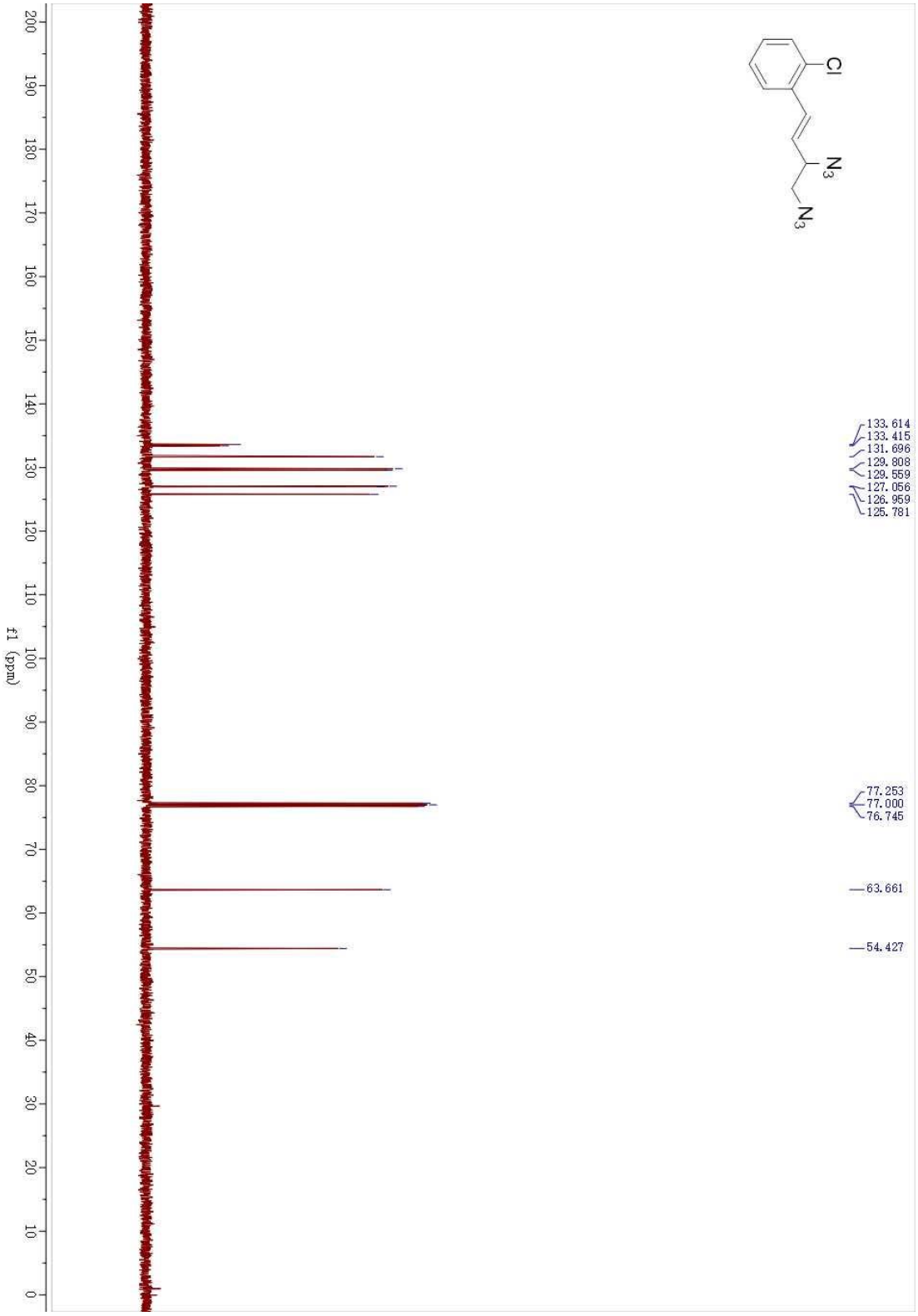
(*E*)-1-chloro-3-(3,4-diazidobut-1-en-1-yl)benzene (4j):



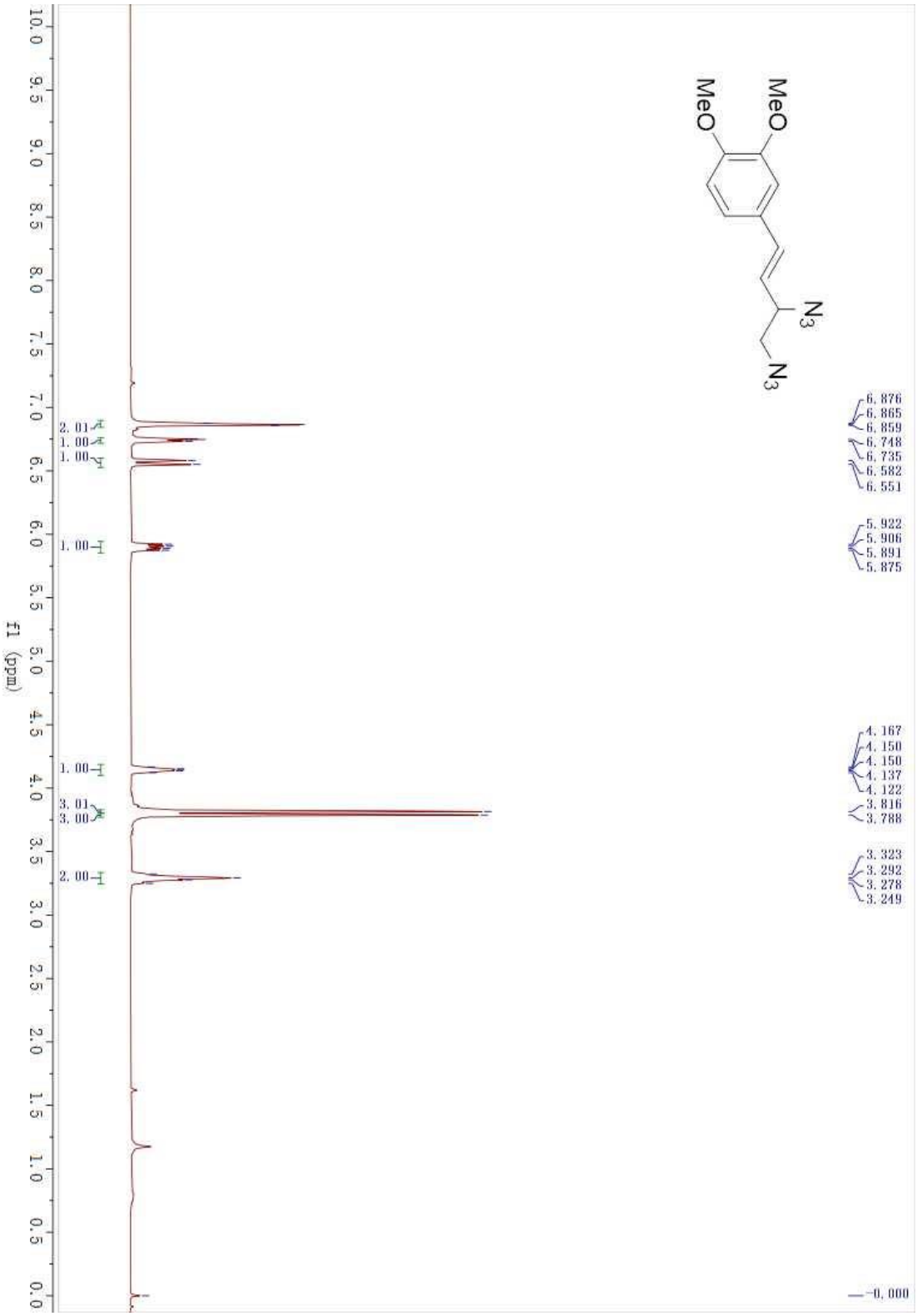

Br
N3
N3
135.424
134.298
133.038
129.791
127.697
127.271
125.945
123.853
77.254
77.000
76.746
63.525
54.397
f1 (ppm)

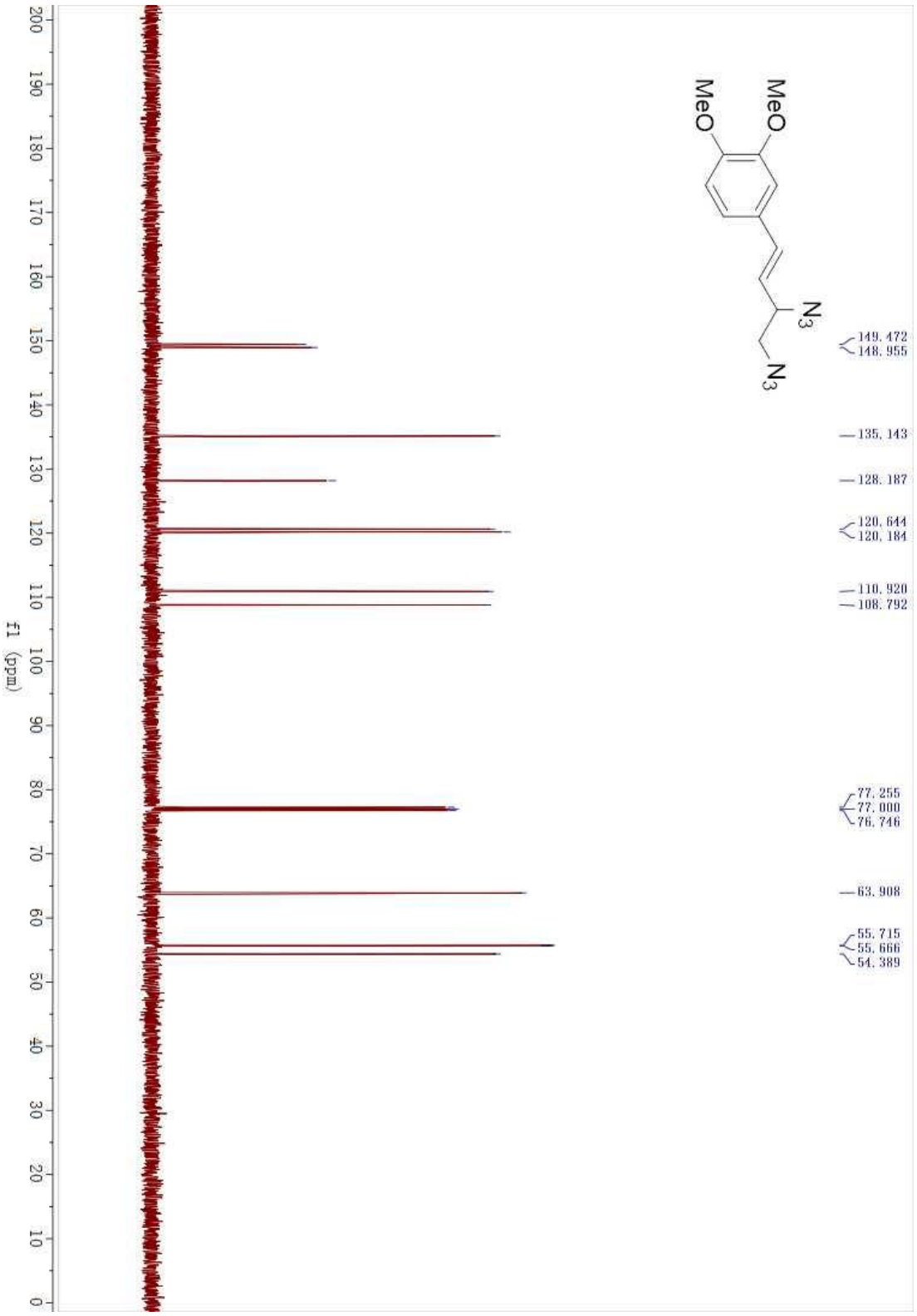
(*E*)-1-chloro-2-(3,4-diazidobut-1-en-1-yl)benzene (4k):



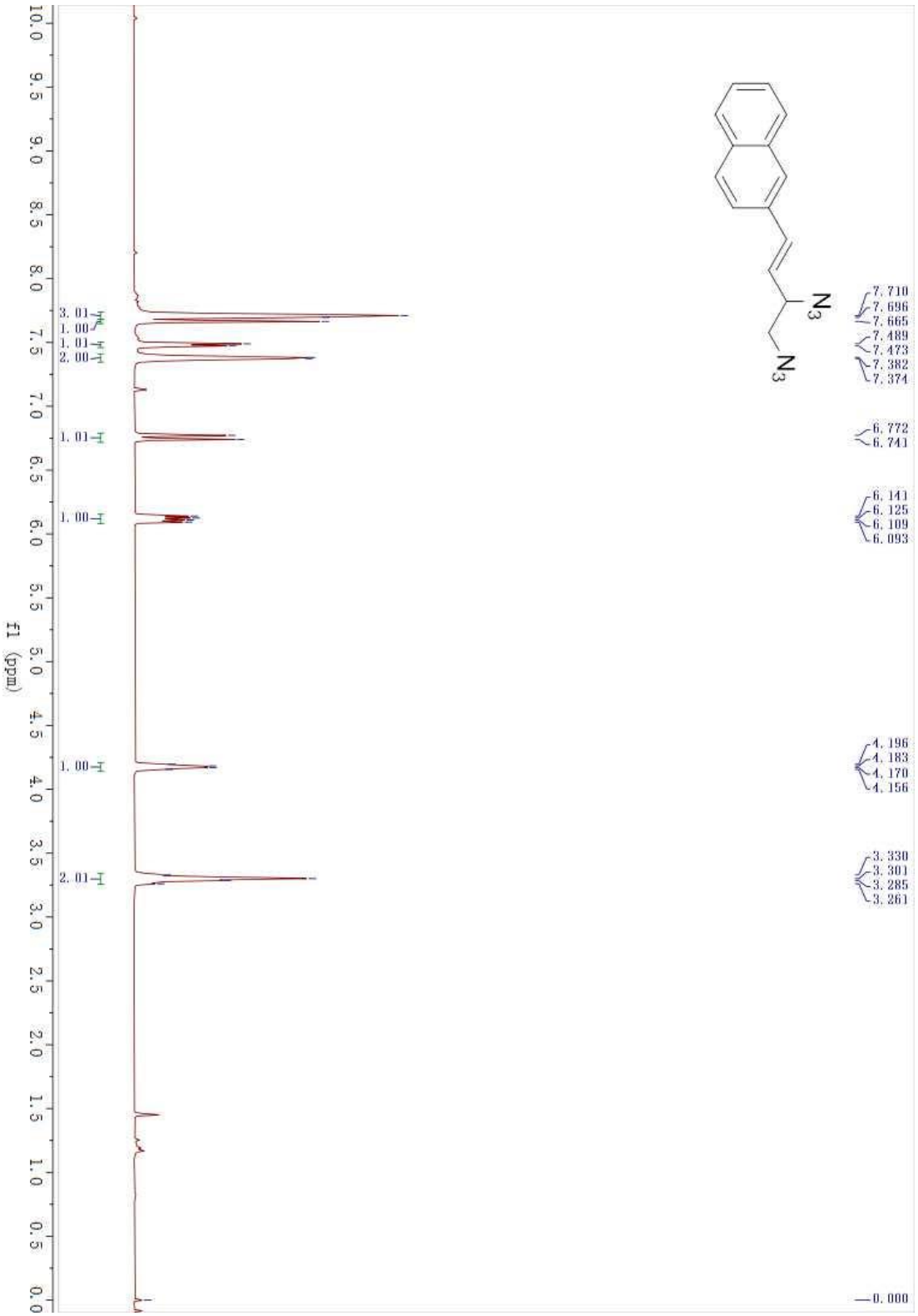

Cl
N3
N3
133.614
133.415
131.696
129.808
129.559
127.056
126.959
125.781
77.253
77.000
76.745
63.661
54.427
f1 (ppm)

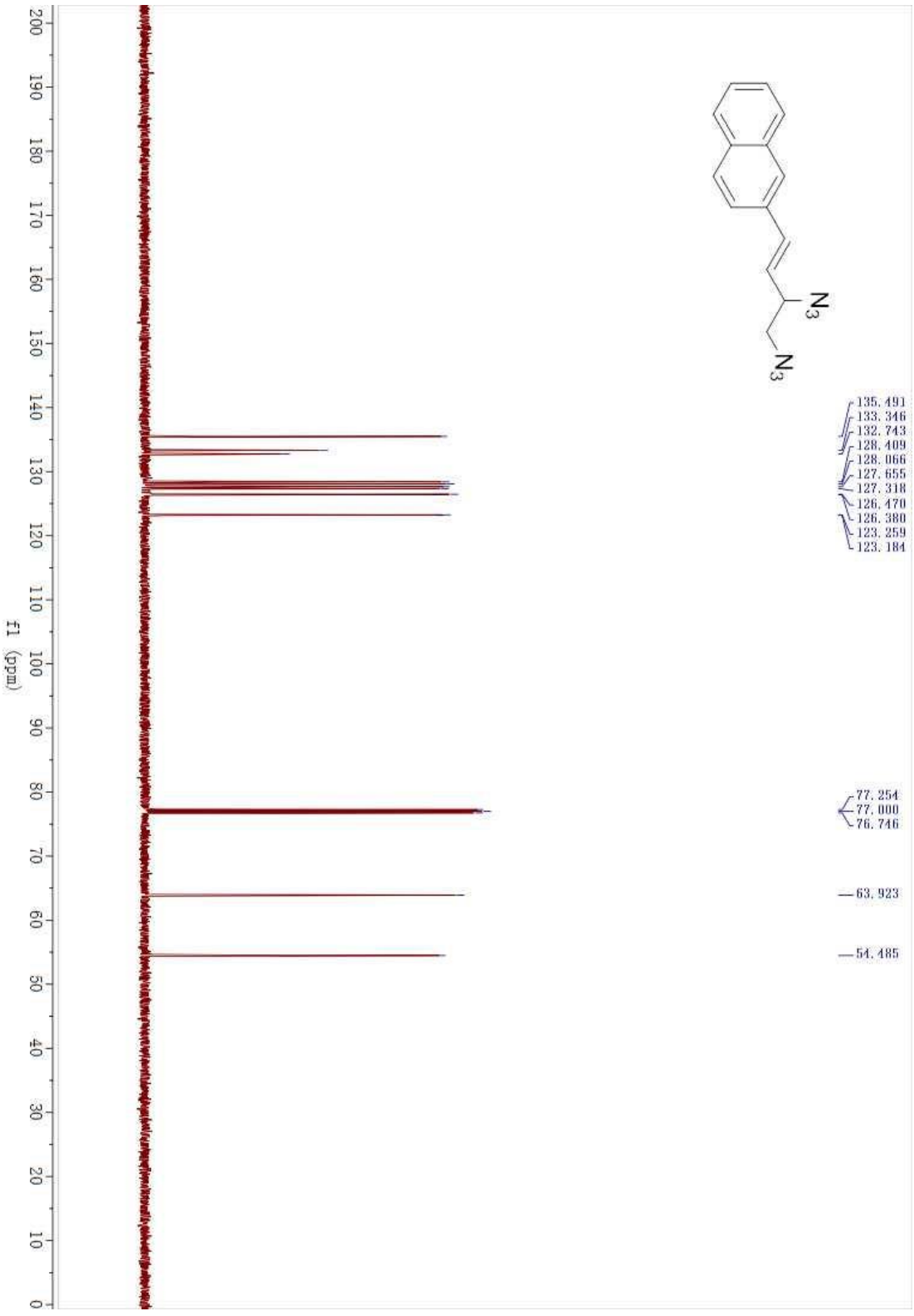
(*E*)-4-(3,4-diazidobut-1-en-1-yl)-1,2-dimethoxybenzene (4l):



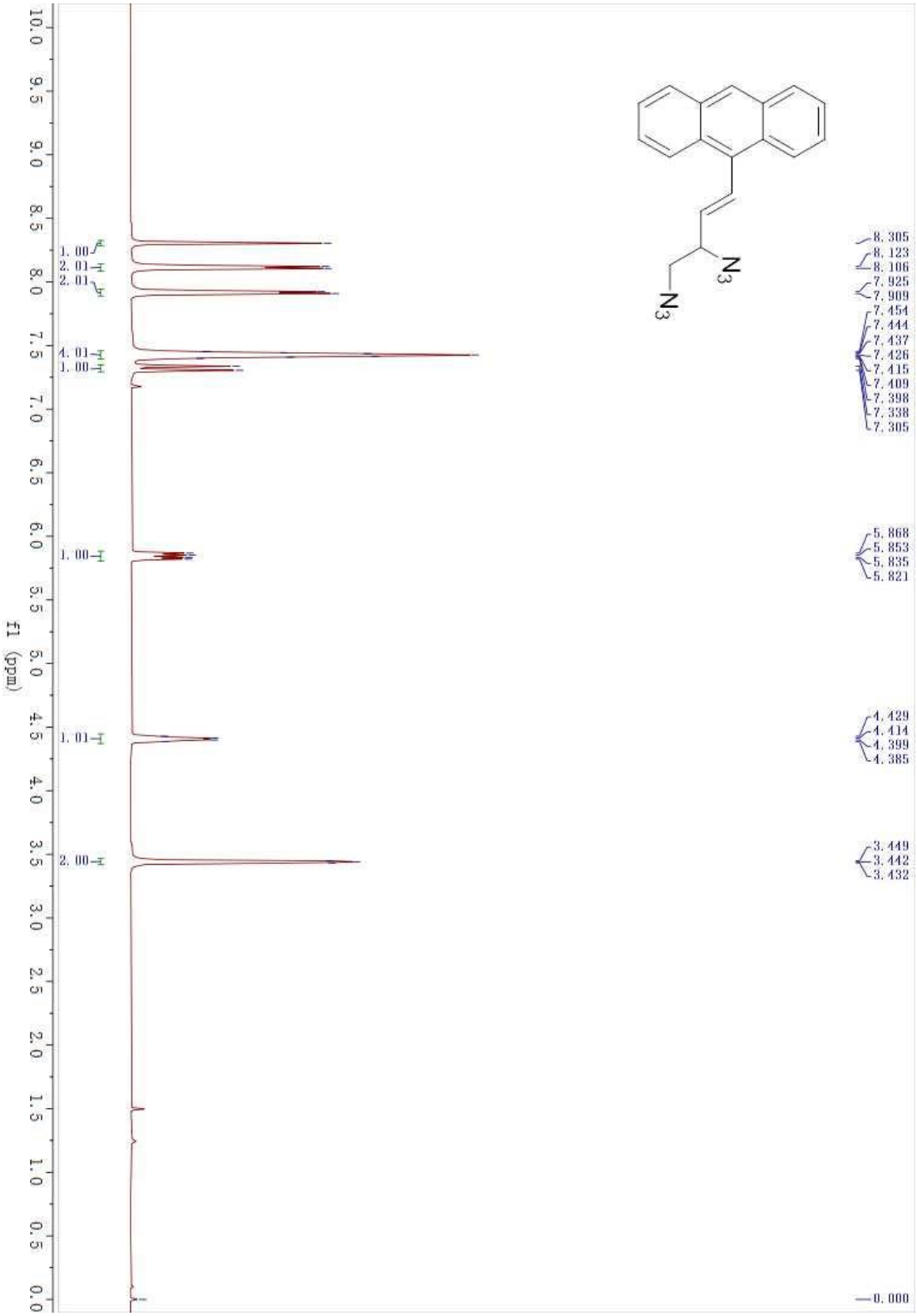

MeO
MeO
N3
N3
149.472
148.955
135.143
128.187
120.644
120.184
110.920
108.792
77.255
77.000
76.746
63.908
55.715
55.666
54.389
f1 (ppm)

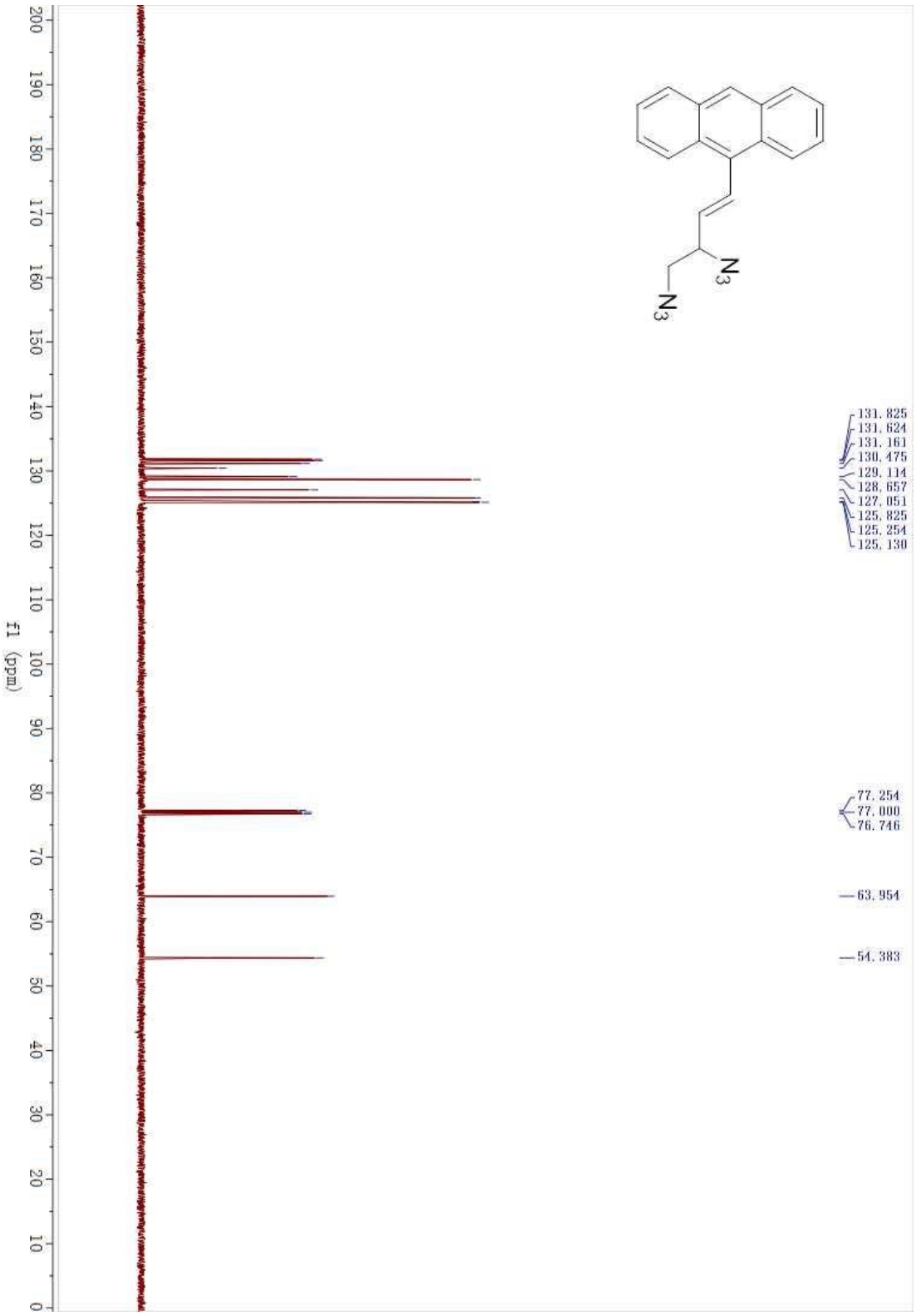
(*E*)-2-(3,4-diazidobut-1-en-1-yl)naphthalene (4m):



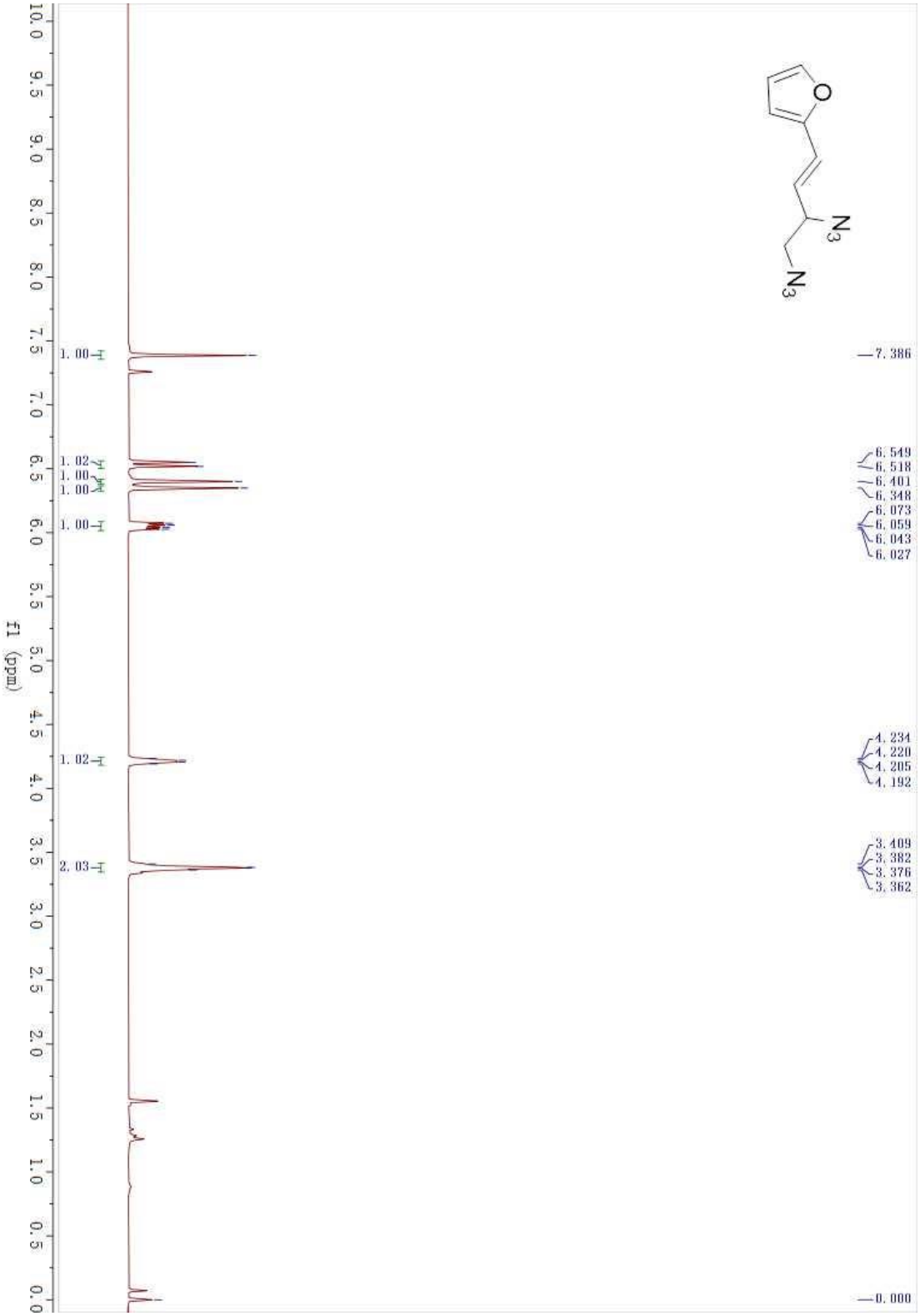

135.491
133.346
132.743
128.409
128.066
127.655
127.318
126.470
126.380
123.259
123.184
77.254
77.000
76.746
63.923
54.485
f1 (ppm)

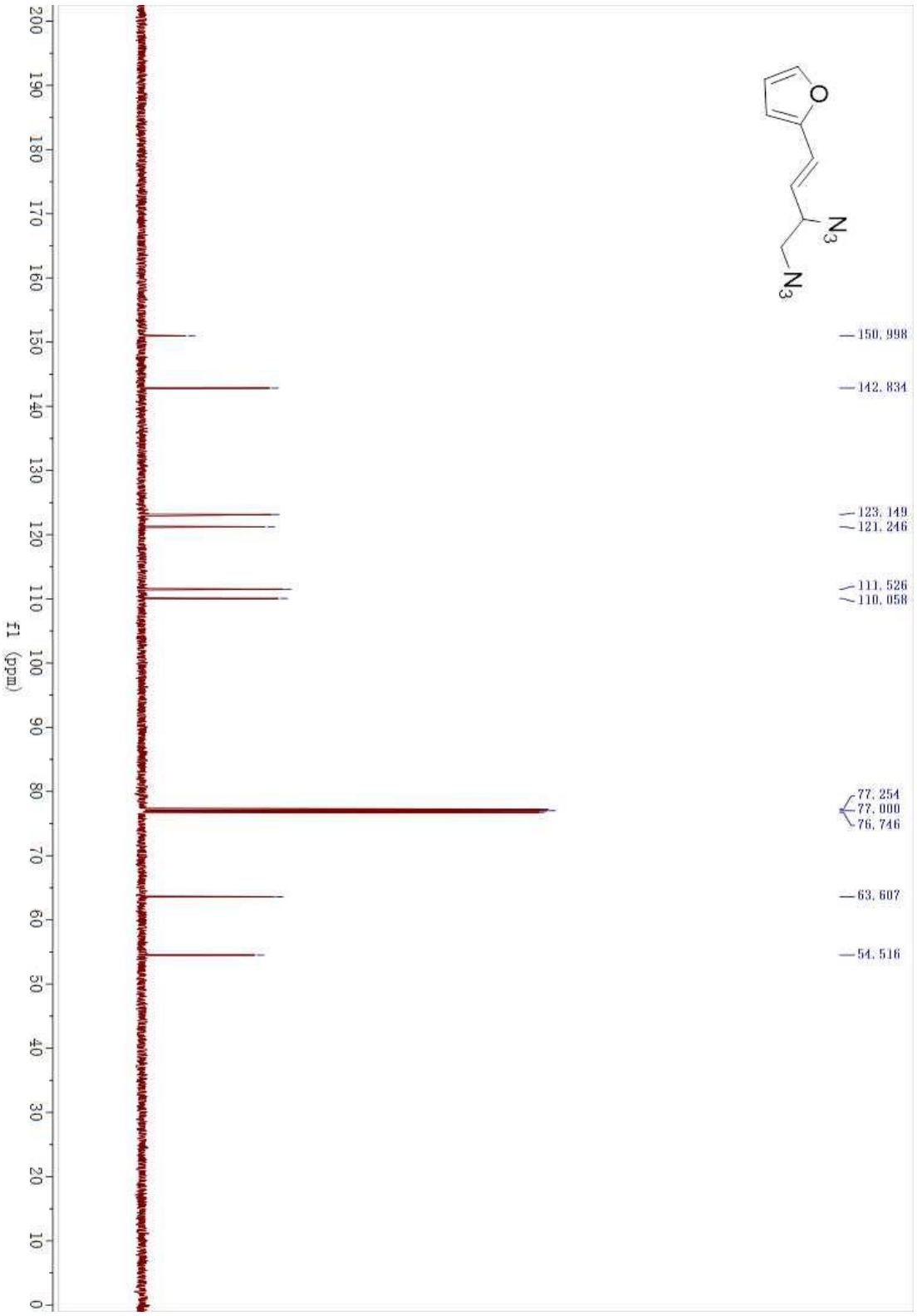
(*E*)-9-(3,4-diazidobut-1-en-1-yl)anthracene (4n):



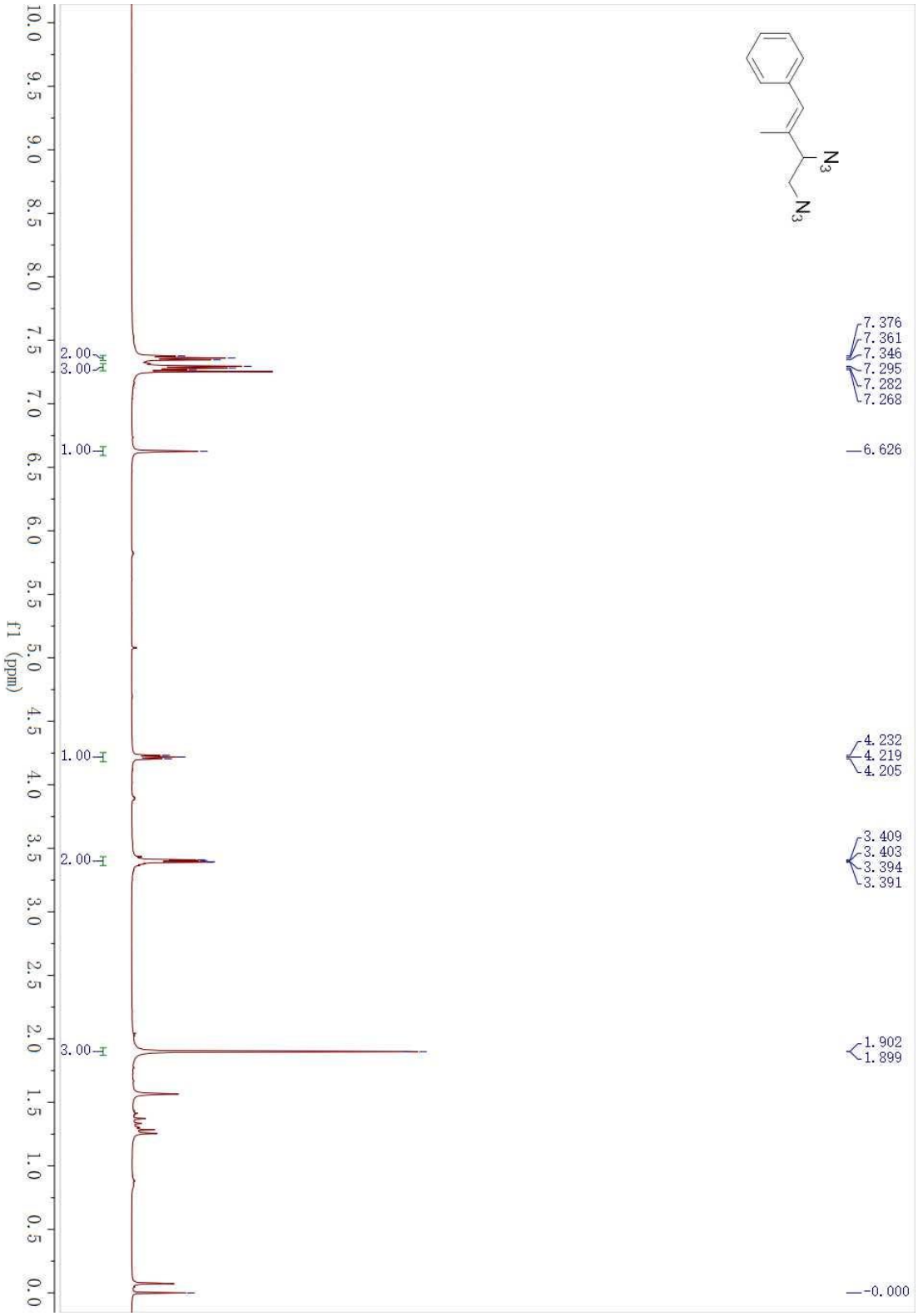

131.825
131.624
131.161
130.475
129.114
128.657
127.051
125.825
125.254
125.130
77.254
77.000
76.746
63.954
54.383
f1 (ppm)

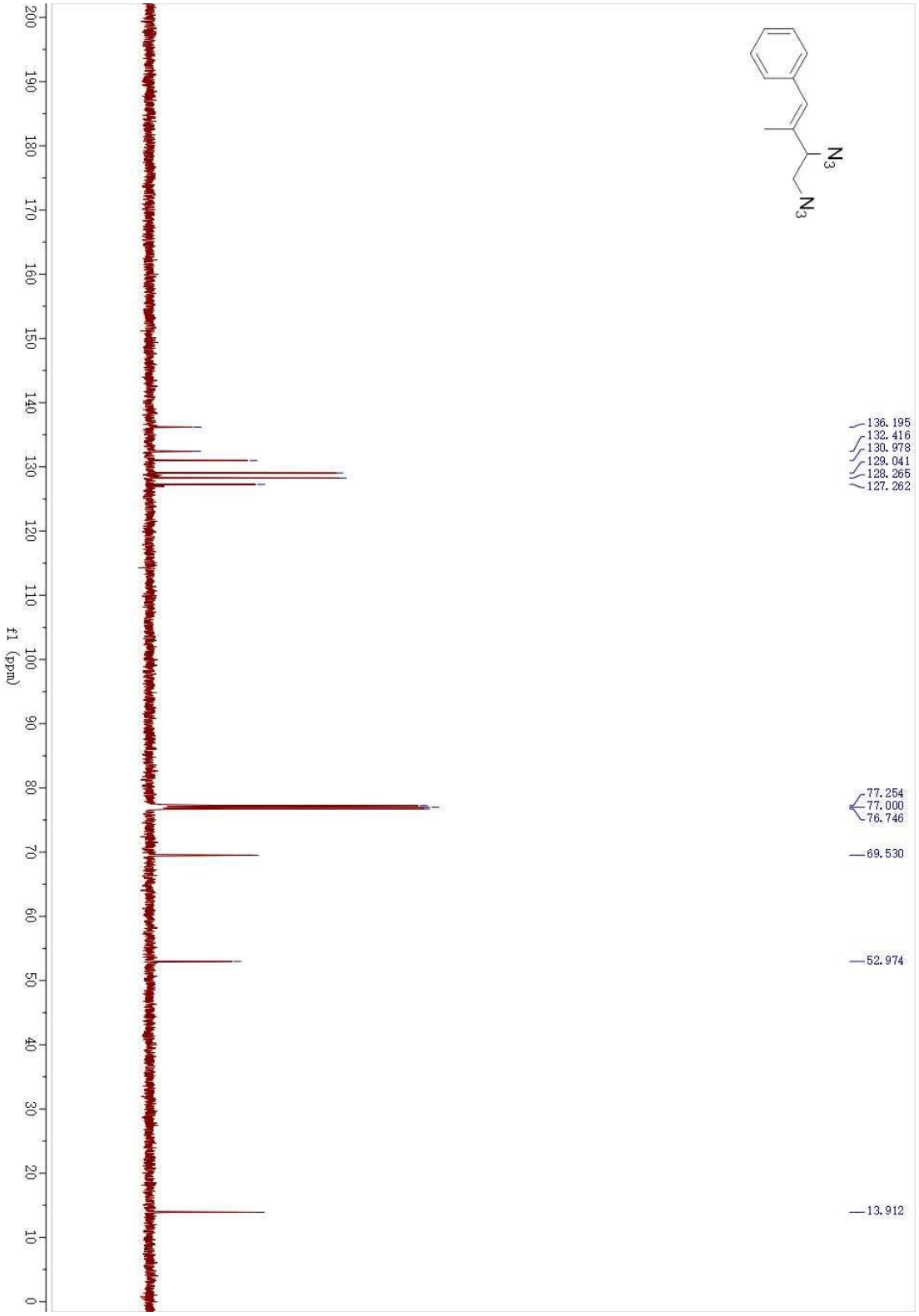
(*E*) -2-(3,4-diazidobut-1-en-1-yl)furan (4o):



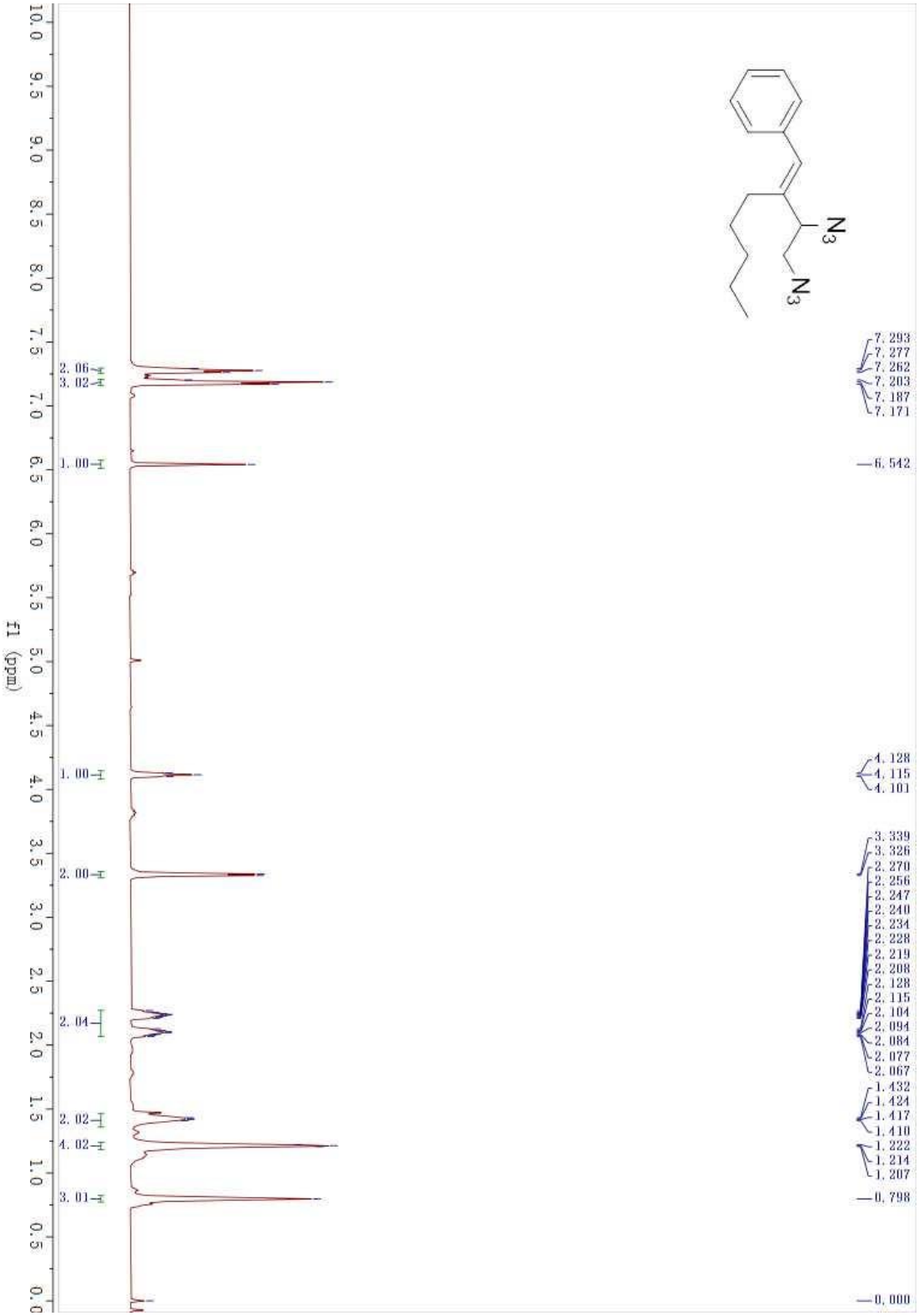

150.998
142.834
123.149
121.246
111.526
110.058
77.254
77.000
76.746
63.607
54.516
f1 (ppm)

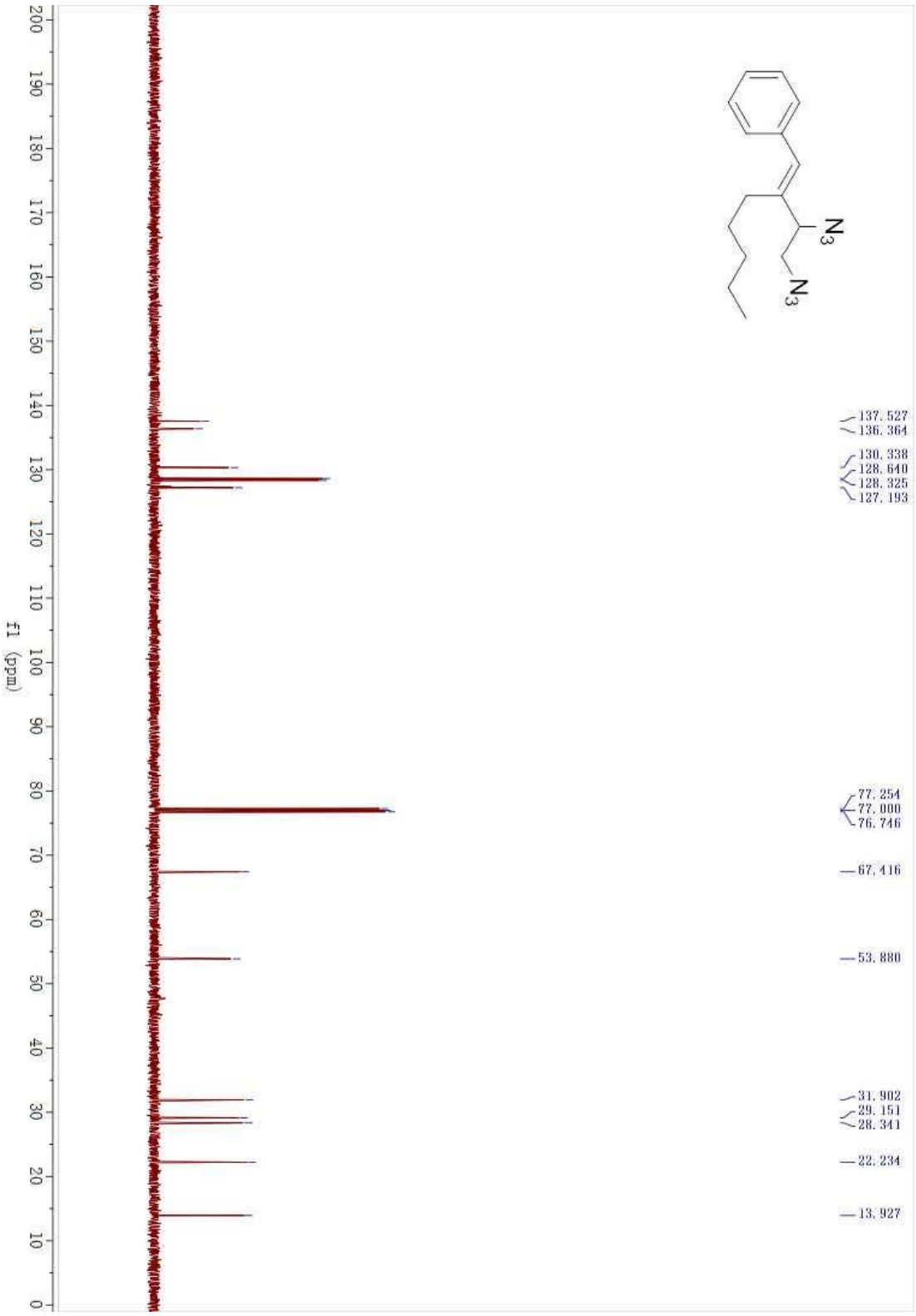
(*E*)-(3,4-diazido-2-methylbut-1-en-1-yl)benzene (4p):



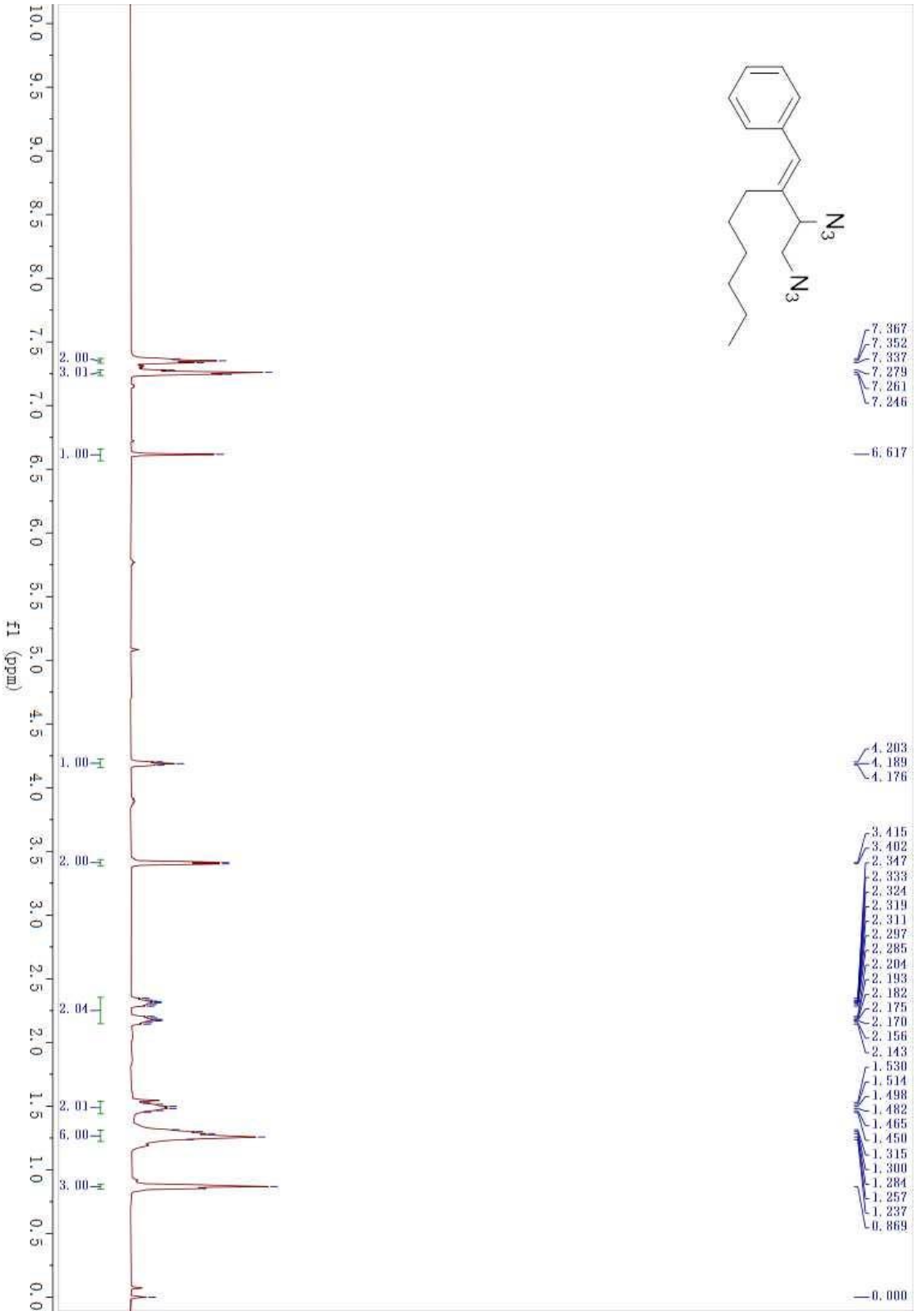

f1 (ppm)
136.195
132.416
130.978
129.041
128.265
127.262
77.254
77.000
76.746
69.530
52.974
13.912

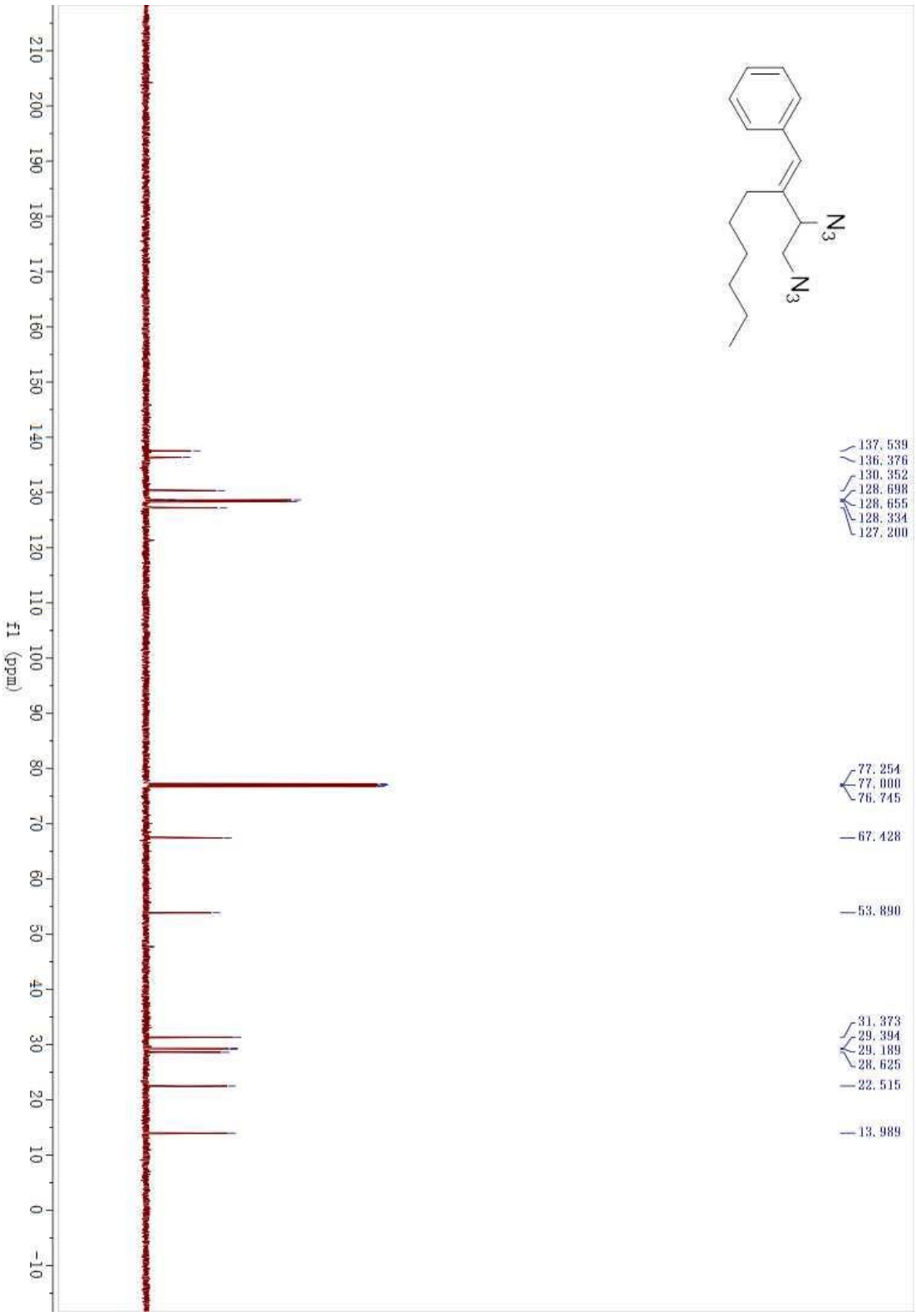
(*E*)-(2-(1,2-diazidoethyl)hept-1-en-1-yl)benzene (4q):



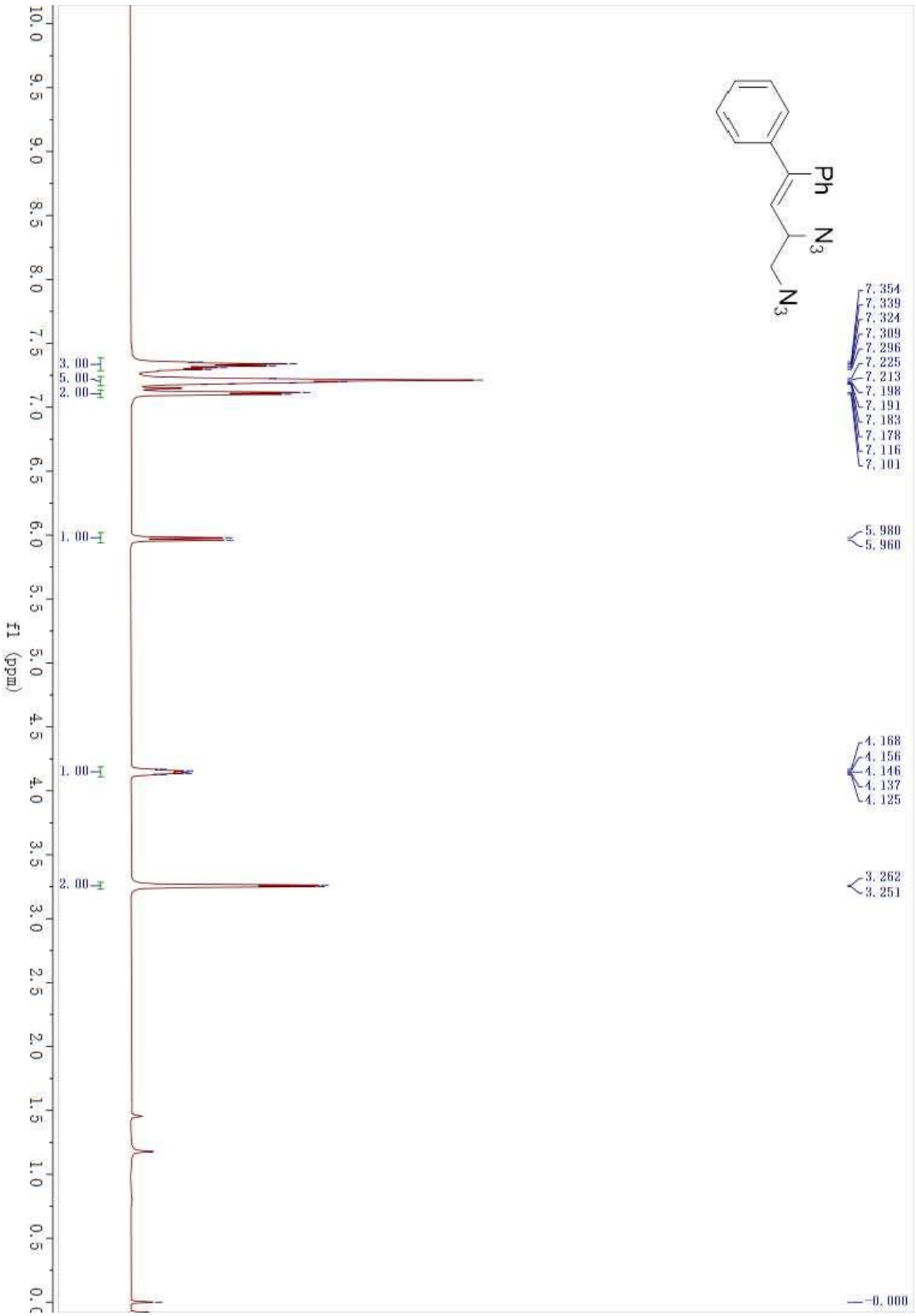

137.527
136.364
130.338
128.640
128.325
127.193
77.254
77.000
76.746
67.416
53.880
31.902
29.151
28.341
22.234
13.927
f1 (ppm)

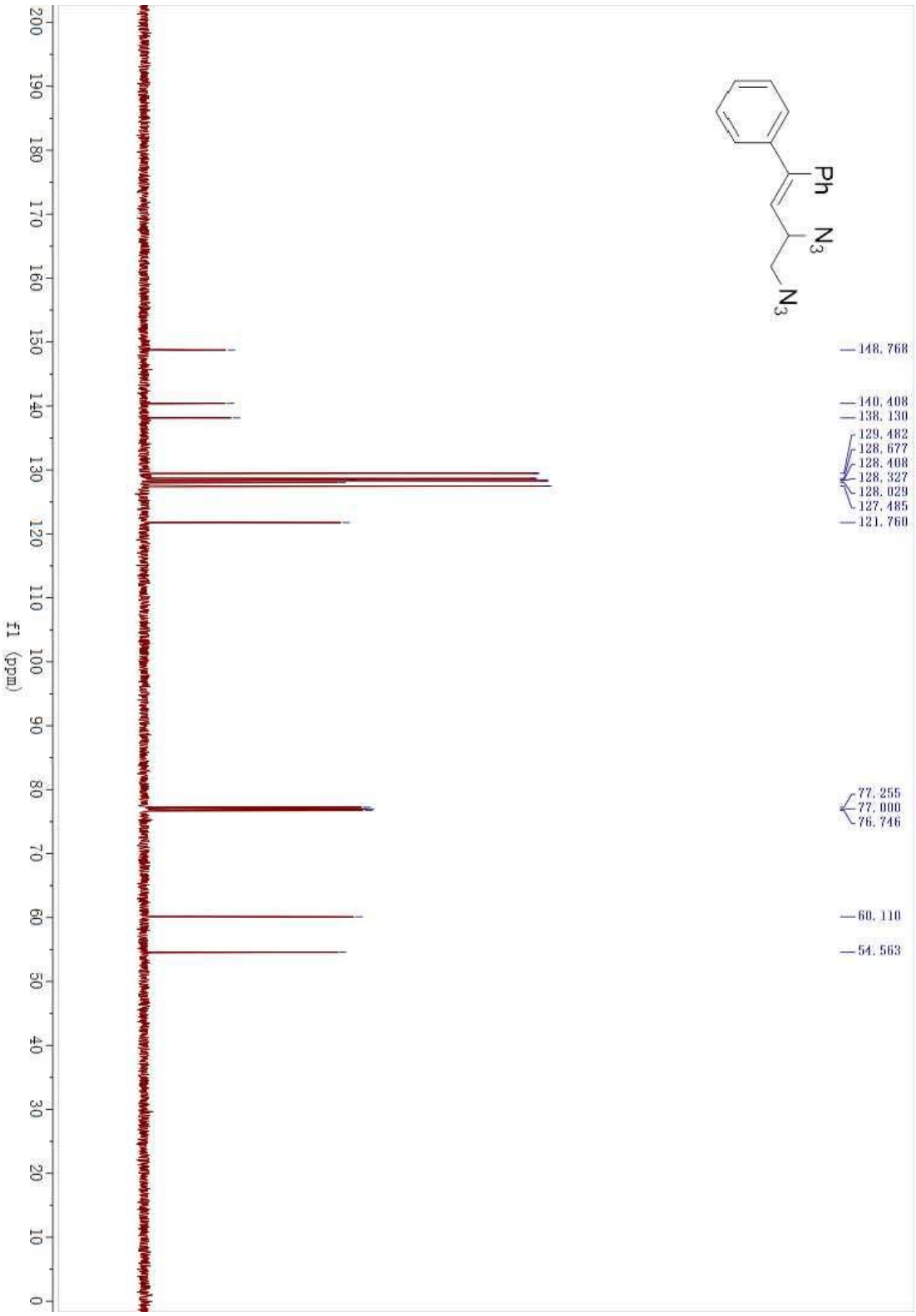
(*E*)-(2-(1,2-diazidoethyl)oct-1-en-1-yl)benzene (4r):



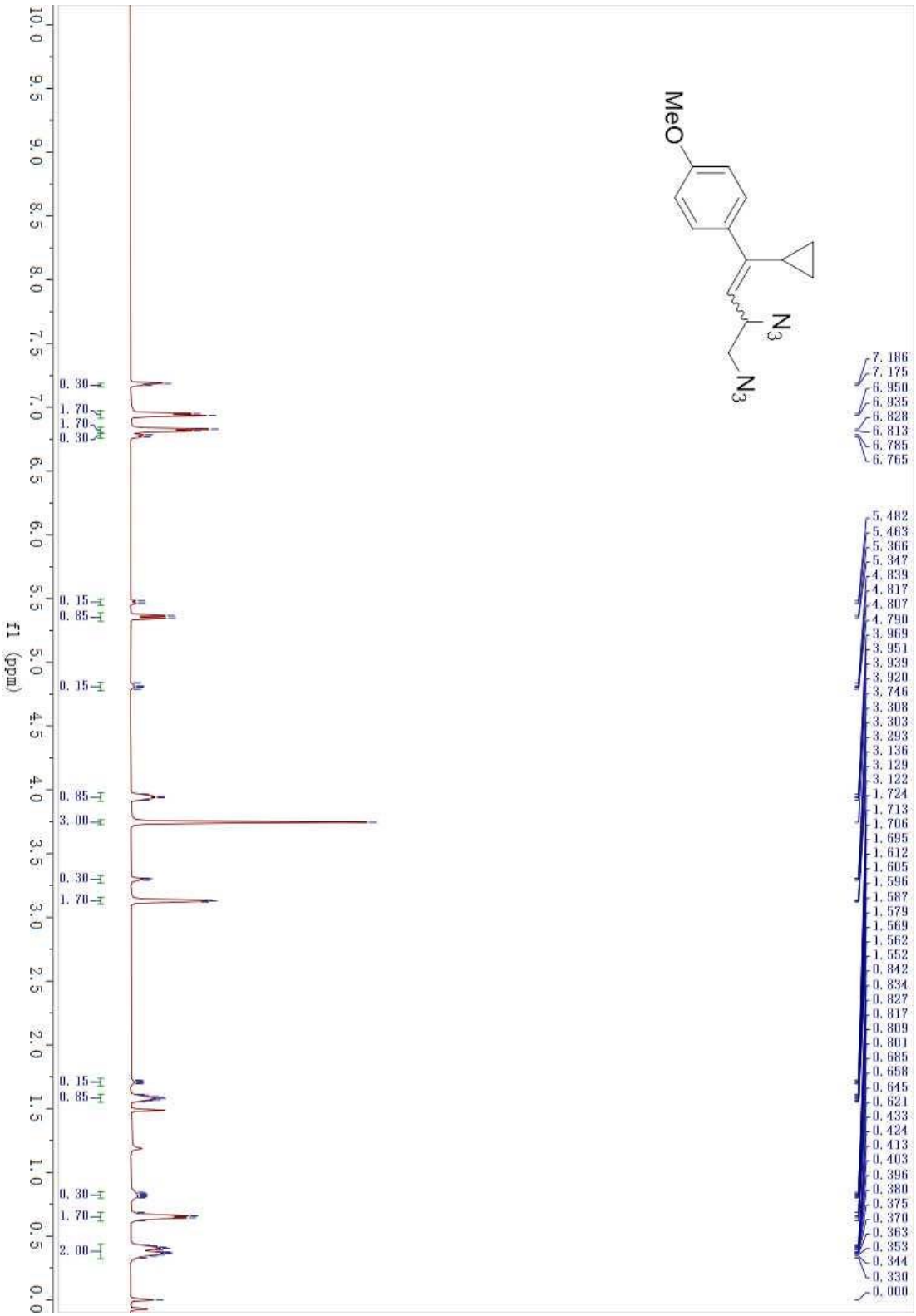

137.539
136.376
130.352
128.698
128.655
128.334
127.200
77.254
77.000
76.745
67.428
53.890
31.373
29.394
29.189
28.625
22.515
13.989
f1 (ppm)

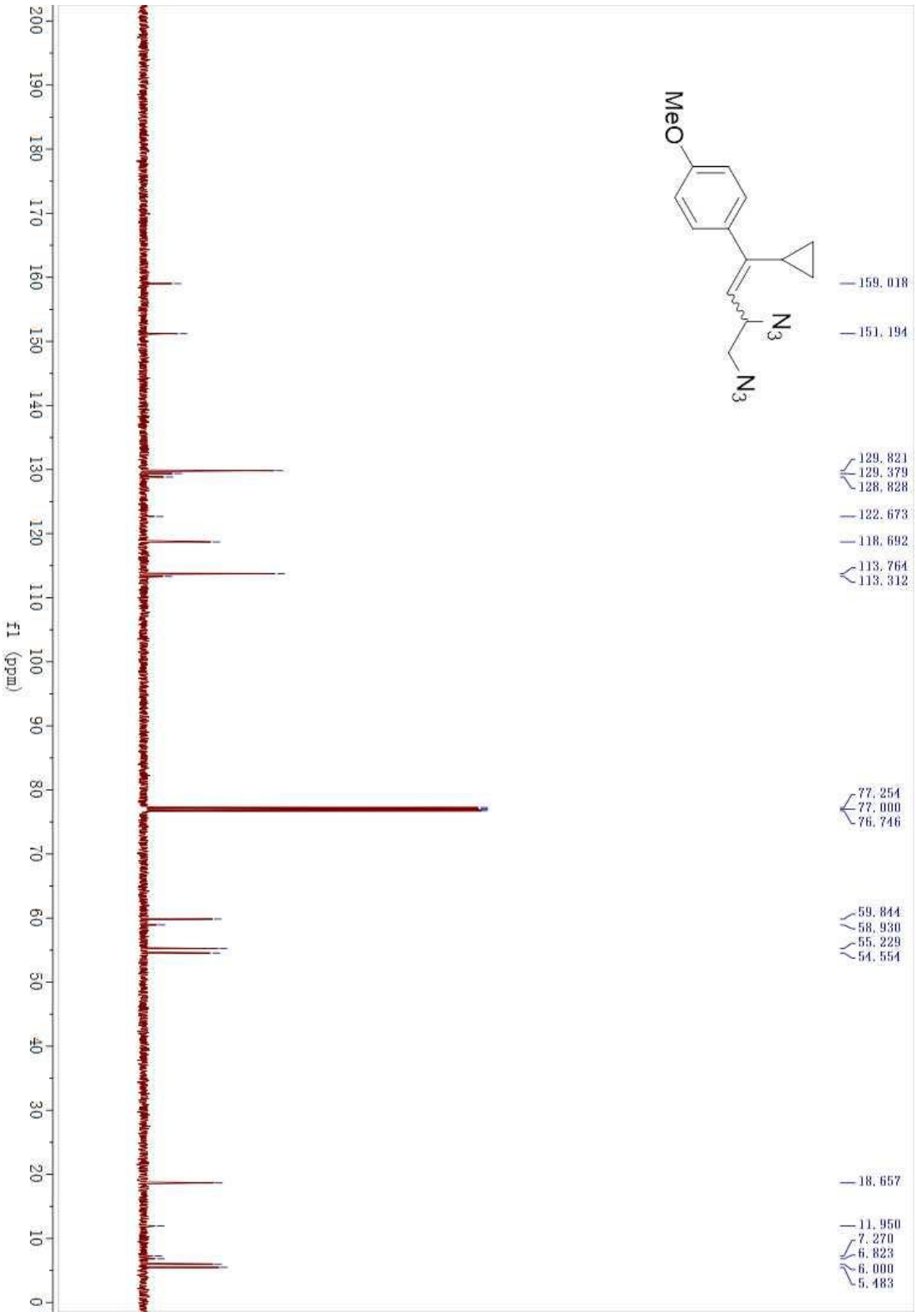
(3,4-diazidobut-1-ene-1,1-diyl)dibenzene (4s):



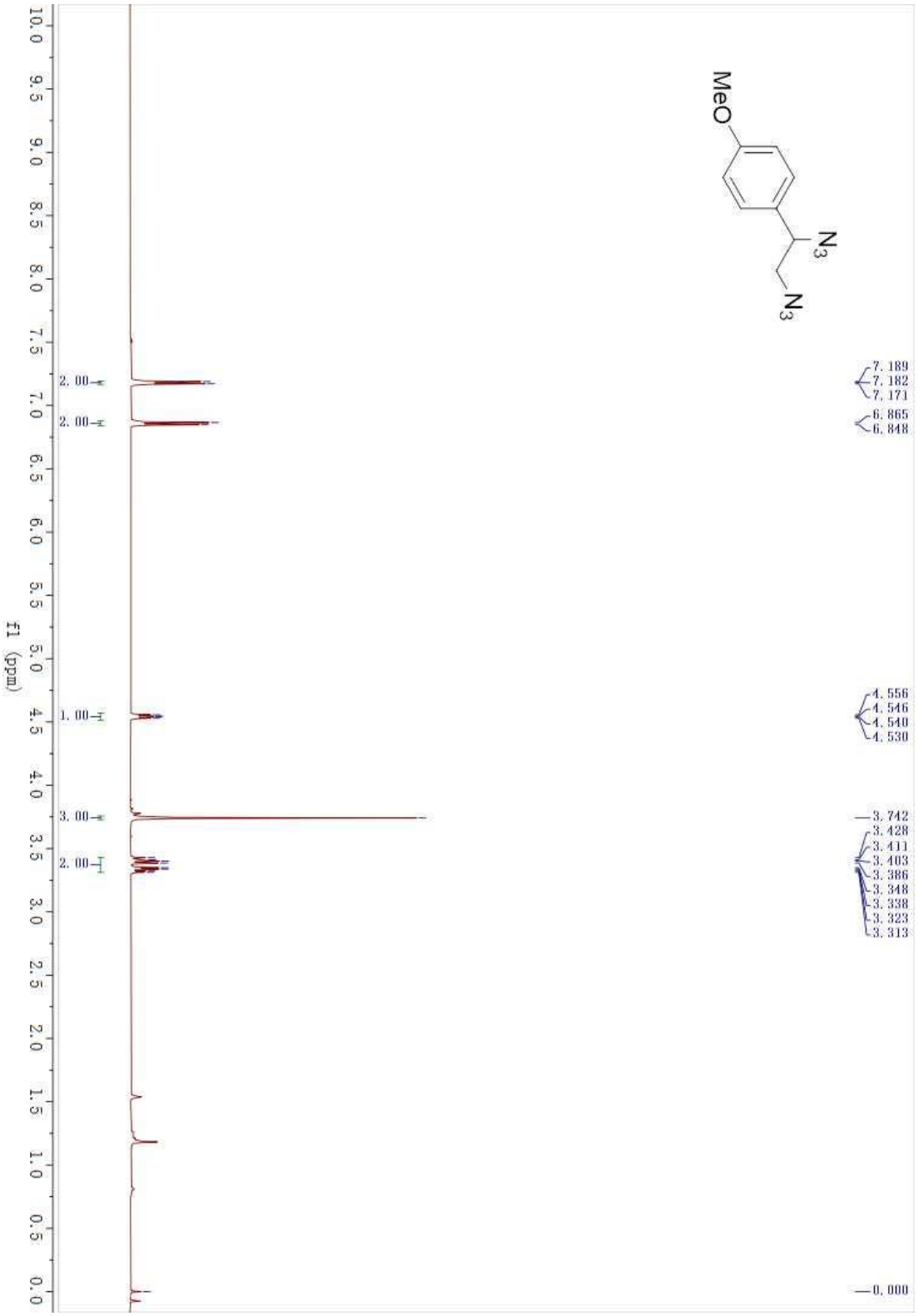

Ph
N3
N3
148.768
140.408
138.130
129.482
128.677
128.408
128.327
128.029
127.485
121.760
77.255
77.000
76.746
60.110
54.563
f1 (ppm)

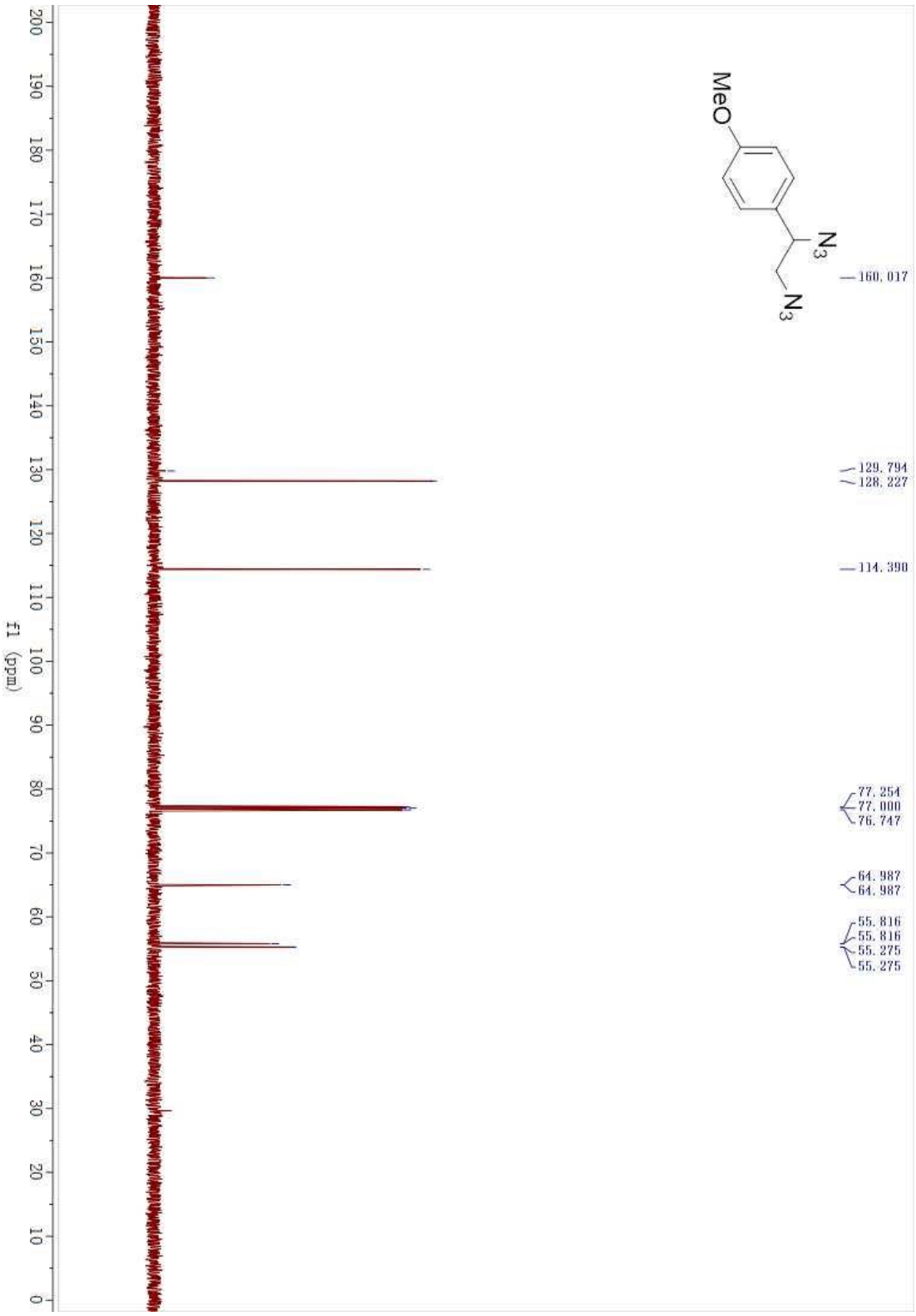
(*E*)-1-(3,4-diazido-1-cyclopropylbut-1-en-1-yl)-4-methoxybenzene (4t):



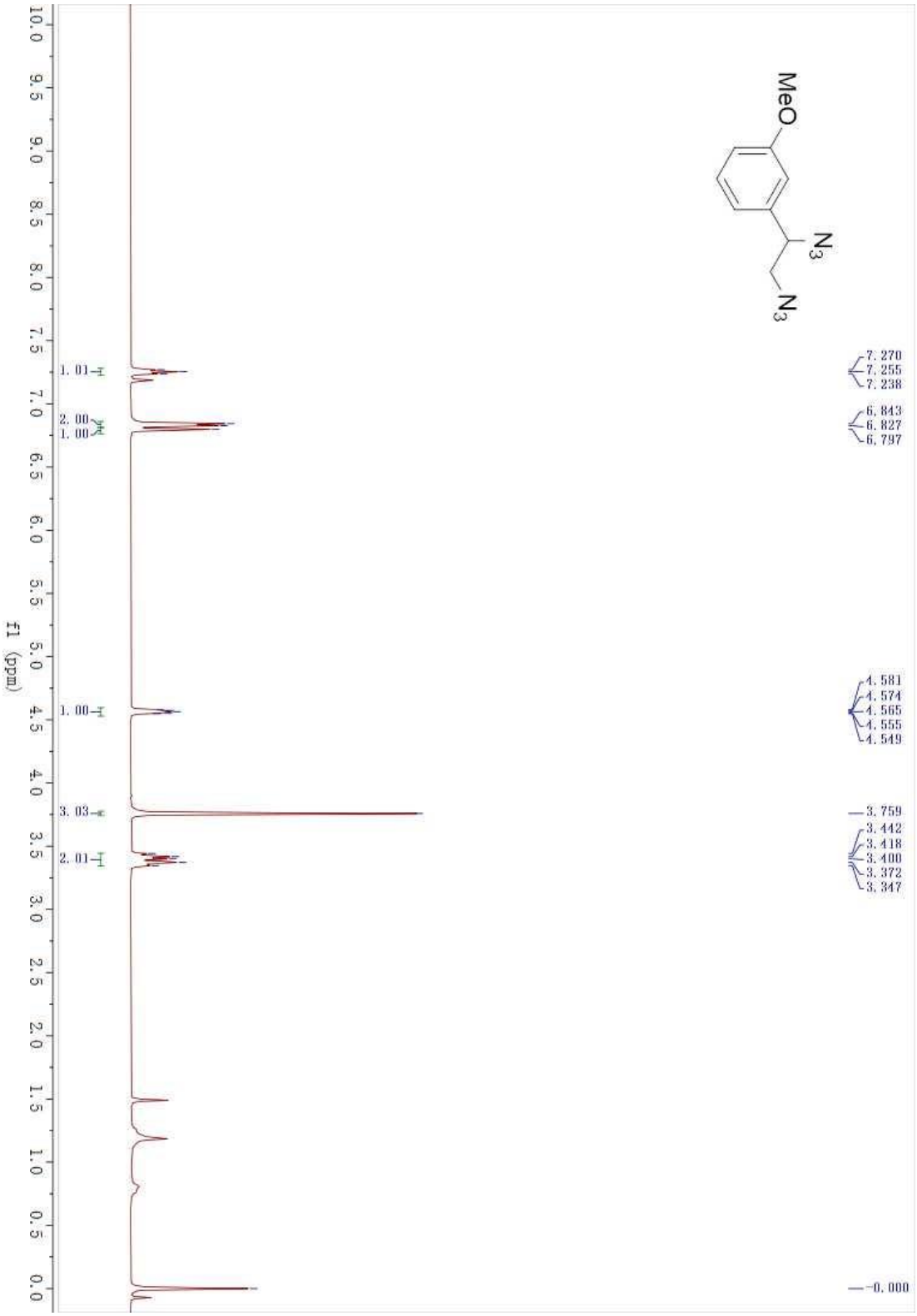

MeO
N3
N3
159.018
151.194
129.821
129.379
128.828
122.673
118.692
113.764
113.312
77.254
77.000
76.746
59.844
58.930
55.229
54.554
18.657
11.950
7.270
6.823
6.000
5.483
f1 (ppm)

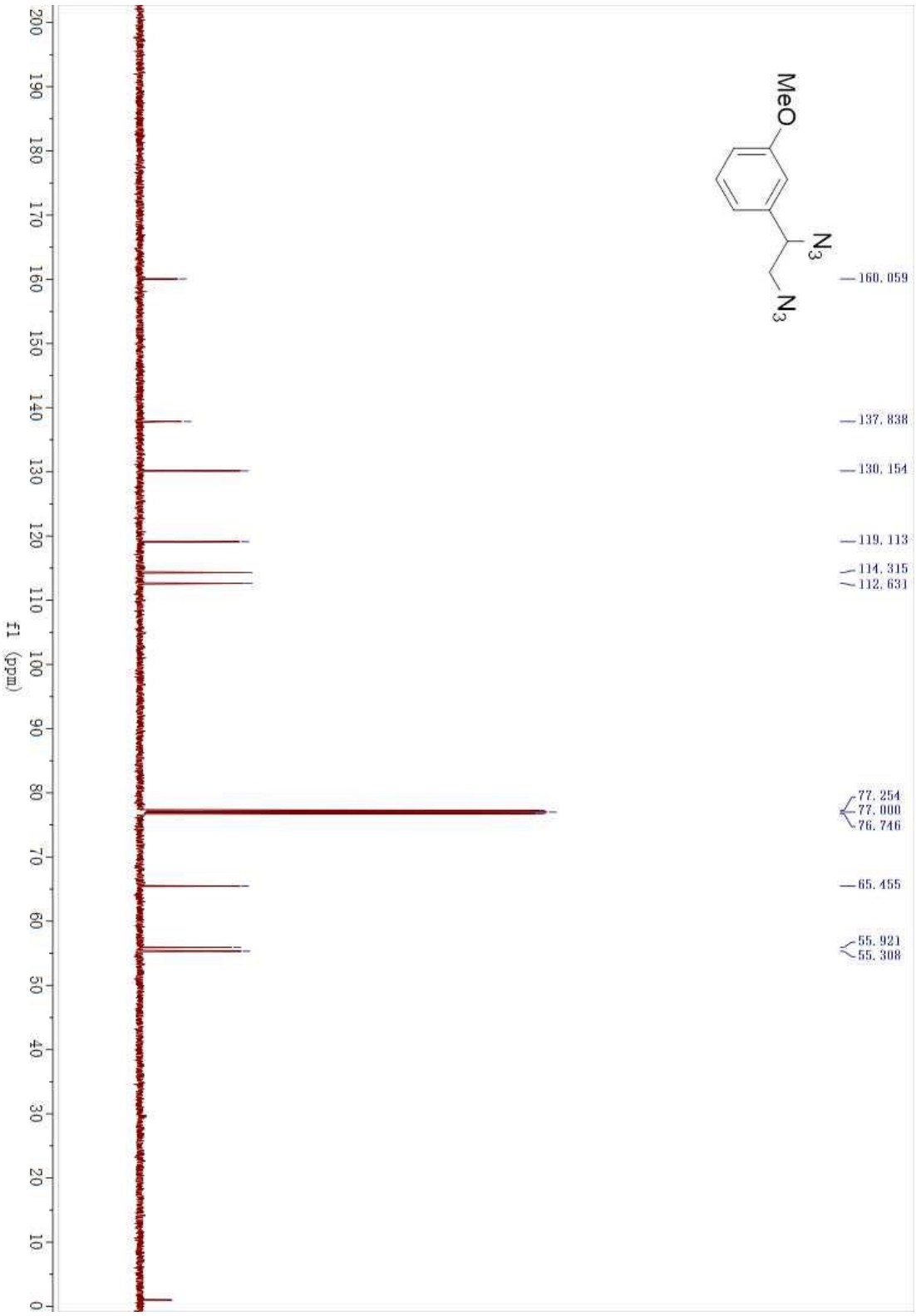
1-(1,2-diazidoethyl)-4-methoxybenzene (4u):



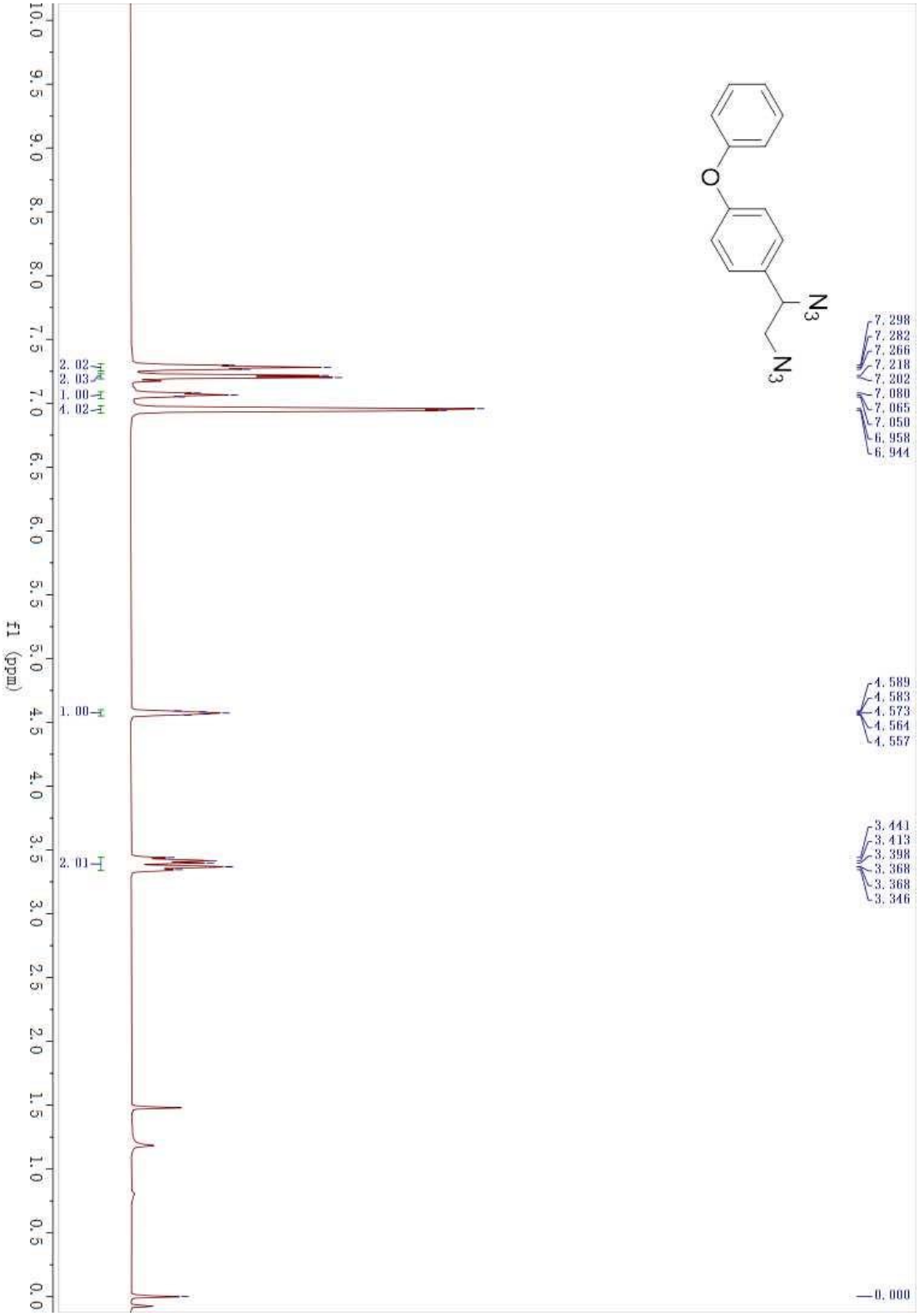

MeO
N3
N3
160.017
129.794
128.227
114.390
77.254
77.000
76.747
64.987
64.987
55.816
55.816
55.275
55.275
f1 (ppm)

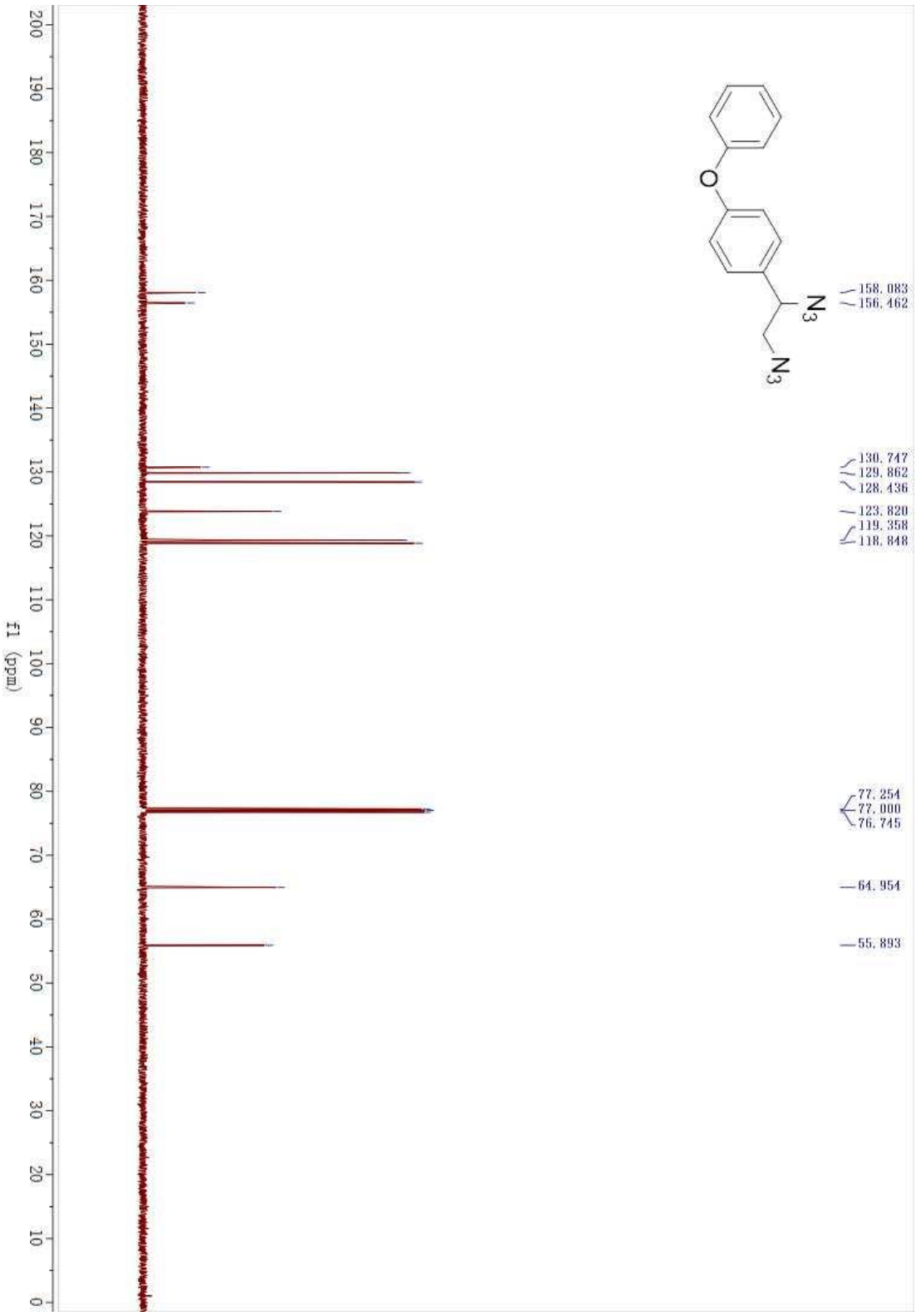
1-(1,2-diazidoethyl)-3-methoxybenzene (4v):



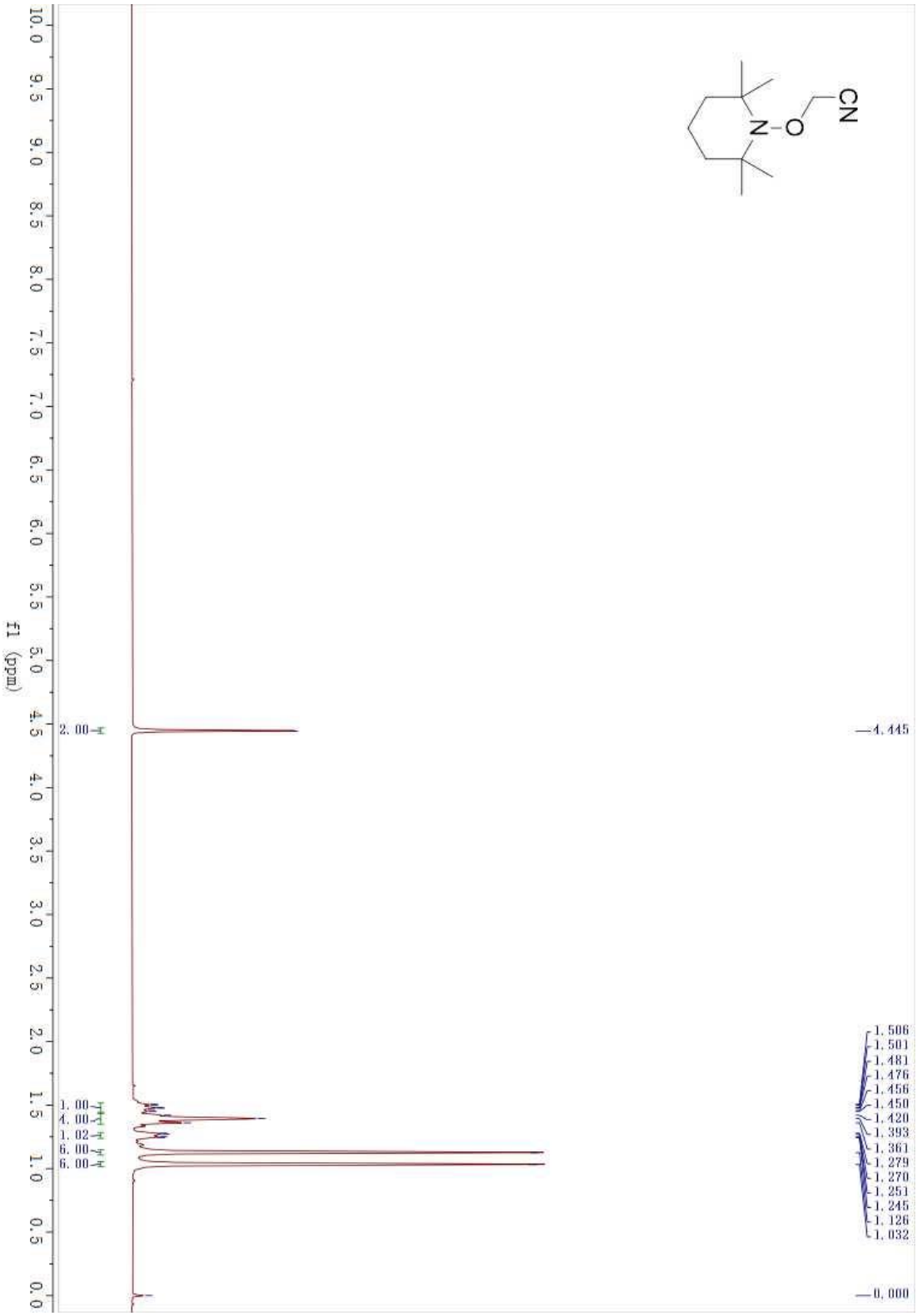

MeO
N3
N3
160.059
137.838
130.154
119.113
114.315
112.631
77.254
77.000
76.746
65.455
55.921
55.308
f1 (ppm)

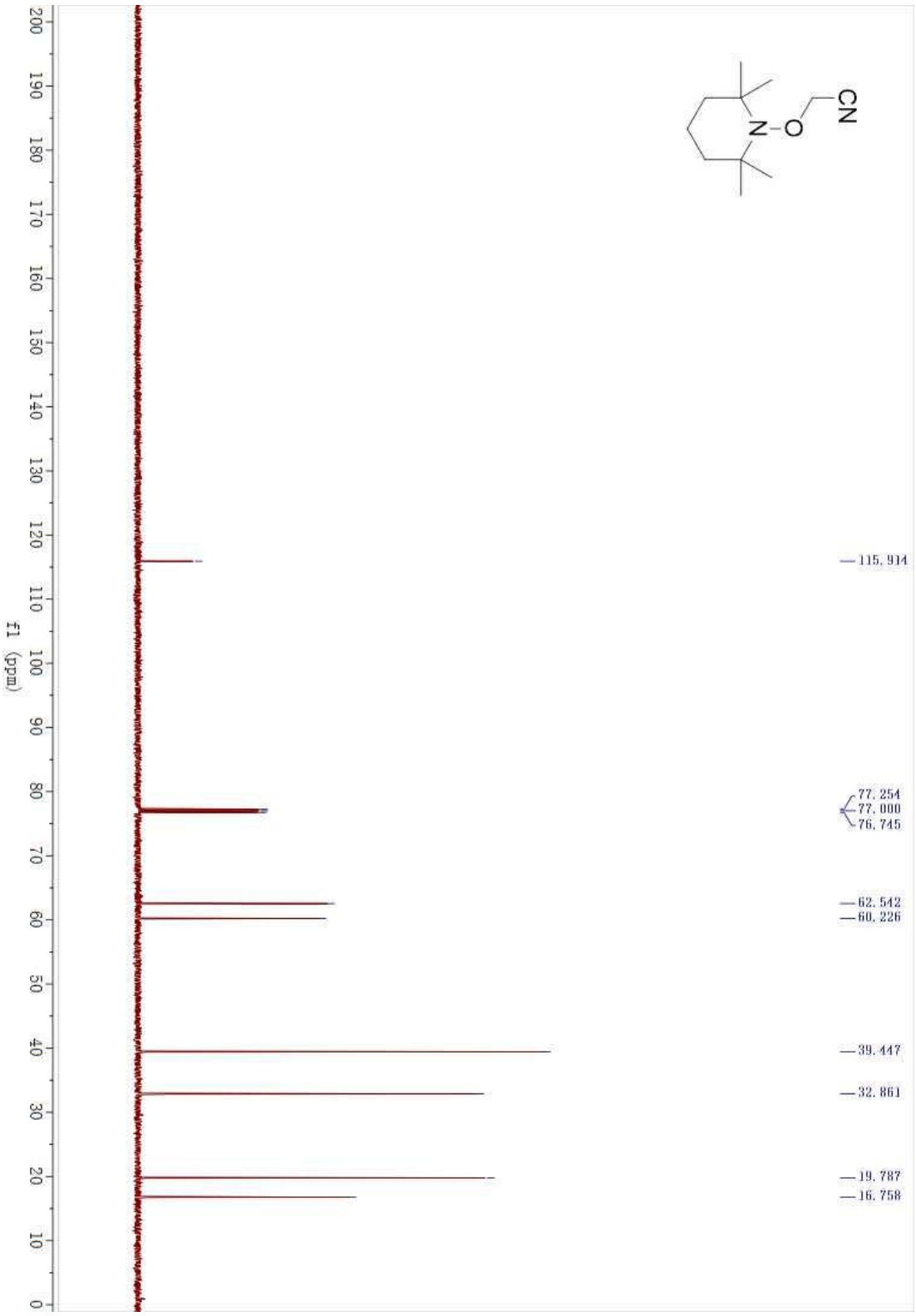
1-(1,2-diazidoethyl)-4-phenoxybenzene (4w):




158.083
156.462
130.747
129.862
128.436
123.820
119.358
118.848
77.254
77.000
76.745
64.954
55.893
f1 (ppm)

2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)acetonitrile:




f1 (ppm)
115.914
77.254
77.000
76.745
62.542
60.226
39.447
32.861
19.787
16.758
N
O
CN