Regio- and Stereoselective Synthesis of α-hydroxy-β-azido tetrazoles

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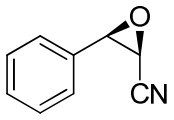
Supplementary material

General Remarks

Column chromatography were performed on a silica gel 230-400 mesh by using various mixtures of dichloromethane (DCM), ethyl acetate (EtOAc), methanol (MeOH), acetic acid (AcOH) and petroleum ether (PE). TLC's were run on Kieselgel $60F_{254}$ plates and revealed by UV light and potassium permanganate (epoxides) or ninhydrin (azido tetrazoles).^{1}H and ^{13}C NMR spectra were collected on a Bruker Avance spectrometer respectively at 200 or 300 MHz and 75 MHz. Data are presented as follows: chemical shift (in ppm on the δ scale relative to δTMS = 0), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet, b = broad), coupling constant (J/Hz), integration and attribution. High resolution mass spectra (HR-MS) were obtained on a Waters Micromass Q-TofMicro instrument. I.R spectra were collected on a NICOLET iS 10(SMART iTR diamond ATR) apparatus. Melting points are uncorrected.

General procedure for the synthesis of cyanoepoxides:

A solution ofstarting aldehydeor ketone(1 mol equiv.)and chloroacetonitrile (1.2 equiv.) in THF (15 mL/10 mmol) was added dropwise to a suspension of freshly crushed NaOH (3 equiv.) in THF (5 mL/10 mmol of NaOH).The reaction was stirred at room temperature and followed by TLC until full conversion of the aldehyde or ketone. Water (100 mL) and dichloromethane (100 mL) were added to the reaction mixture and the organic layer was washed with brine. The organic layer was concentrated under reduced pressure and the crude residue was purified by flash chromatography on silica gel or alumina (compound **9** and **9'**).



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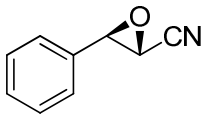


3

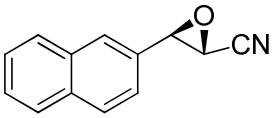
Cis and Trans 3-phenyloxirane-2-carbonitrile

Epoxides **1** and **3** were synthesized following general procedure starting from 3 g of benzaldehyde (28.3mmol) and purified by chromatography on silica gel using PE/EtOAc 50:1 as eluant.Pure *trans* and *cis* isomers were isolated as colorlessoils crystallising on standing (*trans*: 1.2g, 29% yield, *cis*: 1.3g, 32% yield).(EtOAc/PE : 1/9,Rf_{cis} = 0.35; Rf_{trans} = 0.50).

Trans stereoisomer: **^{1}H NMR** (200 MHz, $CDCl_3$) δ7.45-7.40 (m, 3H), 7.35-7.25 (m, 2H), 4.30 (d, J=1.8 Hz, 1H), 3.43 (d, J=1.8 Hz, 1H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ132.75, 129.84, 128.99, 125.66, 116.03, 58.49, 44.64.

*Cis*stereoisomer:**^{1}H NMR** (200 MHz, $CDCl_3$) δ7.50-7.35 (m, 5H),4.26 (d, J=3.7 Hz, 1H), 3.79 (d, J=3.7 Hz, 1H).**^{13}C NMR** (75 MHz, $CDCl_3$) δ131.41, 129.73, 128.70, 126.32, 115.07, 57.72, 45.12.

IR (cm^{-1}) ν_{max}: 3047, 2252, 1454, 1387, 1190, 947, 923, 879, 825, 774, 752, 699, 639, 580, 537, 487.**HRMS** (ESI, TOF MS) m/z calculated for [M+Na]+: 168.0426, found: 168.0427.



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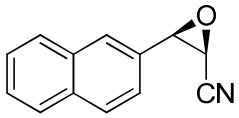


5

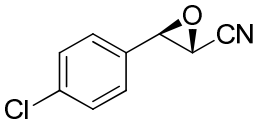
Cis and Trans 3-(naphthalen-2-yl)oxirane-2-carbonitrile

Epoxides **4** and **5** were synthesized following general procedure starting from 1.2 g of *2*-naphthaldehyde (7.7 mmol) and purified by chromatography on silica gel using PE/EtOAc 98:2 as eluant. Pure *trans* and *cis* isomers were isolated as white solids (*trans*: 485 mg, 32% yield, *cis*: 495 mg, 33% yield).(EtOAc/PE: 1/9, Rf_{cis} = 0.20; Rf_{trans}: Rf = 0.45).

Trans stereoisomer: **^{1}H NMR** (200 MHz, $CDCl_3$) δ 7.95-7.80 (m, 4H), 7.60-7.50 (m, 2H), 7.35-7.25 (m, 1H), 4.46 (d, J=1.7 Hz, 1H), 3.52 (d, J=1.7 Hz, 1H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 133.87, 132.93, 130.04, 129.09, 127.98, 127.91, 127.08, 126.98, 126.15, 121.84, 116.06, 58.79, 44.71.

Cis stereoisomer:**^{1}H NMR** (200 MHz, $CDCl_3$) δ 7.80-7.00 (m, 4H), 7.65-7.45 (m, 3H), 4.43 (d, J=3.7 Hz, 1H), 3.87 (d, J=3.7 Hz, 1H).**^{13}C NMR** (75 MHz, $CDCl_3$) δ133.89, 132.88, 128.81, 128.70, 128.18, 127.90, 126.93, 126.76, 126.31, 122.99, 115.06, 57.96, 45.28.

IR (cm^{-1}). ν_{max}: 3049, 2246, 1509, 900, 867, 837, 827, 818, 756, 715, 480, 475. **HRMS** (ESI, TOF MS) m/z calculated for [M+H]+: 196.0762, found: 196.0759.



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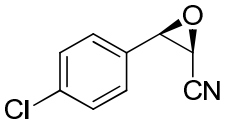


7

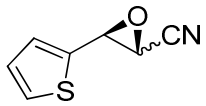
Cis and Trans 3-(4-chlorophenyl)oxirane-2-carbonitrile

Epoxides **6** and **7** were synthesized following general procedure starting from 1.2 g of *p*-chlorobenzaldehyde (8.5mmol) and purified by chromatography on silica gel using PE/EtOAc 98:2 as eluant. Pure *trans* and *cis* isomers were isolated as white solids(*trans*: 213 mg, 14% yield, *cis*: 430 mg, 28% yield).(EtOAc/PE: 1/9, Rf_{cis}= 0.23; Rf_{trans}= 0.45).

Trans stereoisomer: **^{1}H NMR** (200 MHz, $CDCl_3$) δ 7.39 (d, J=8.5 Hz, 2H), 7.23 (d, J=8.5 Hz, 2H), 4.29 (d, J=1.7 Hz, 1H), 3.41 (d, J=1.7 Hz, 1H). **^{13}C NMR** (75 MHz, CDCl3) δ135.85, 131.26, 129.28, 127.01, 115.73, 57.89, 44.66.

Cis stereoisomer:**^{1}H NMR** (200 MHz, $CDCl_3$) δ 7.43 (d, J=8.6 Hz, 2H) 7.37 (d, J=8.6 Hz, 2H), 4.25 (d, J=3.7 Hz, 1H), 3.80 (d, J=3.7 Hz, 1H).**^{13}C NMR** (75 MHz, $CDCl_3$) δ135.74, 129.96, 129.02, 127.71, 114.84, 57.14, 45.11.

IR (cm^{-1}) ν_{max}: 3047, 2246, 1495, 1090, 839, 827, 767, 510**.HRMS** (ESI,TOF MS): not detected.

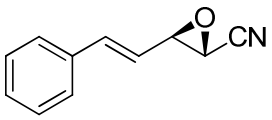


8

3-Thiophen-2-yl-oxirane-2-carbonitrile

Epoxide**8** was synthesized following general procedure starting from 5g of 2-thiophenecarboxaldehyde (44 mmol) and was isolated without purification as abrown liquid (6 g, 89% yield), 1:2 mixture of *cis*/*trans*stereoisomers. (EtOAc/PE: 1/9, Rf_{trans} = 0.62, Rf_{cis} = 0.35).

^{1}H NMR (200 MHz, $CDCl_3$) δ 7.40 (dd, *J* = 5.0, 1.2 Hz, $1H_{cis}$), 7.36 (dd, *J* = 5.0, 0.9 Hz, $1H_{trans}$), 7.29 (dd, *J* = 2.4, 1.9 Hz, $1H_{cis}$), 7.22 (dd, *J* = 3.6, 0.8 Hz, $1H_{trans}$), 7.08 (dd, *J* = 5.4, 4.0 Hz, $1H_{cis}$), 7.04 (dd, *J* = 5.0, 3.6 Hz, $1H_{trans}$), 4.54 (d, *J* = 1.8 Hz, $1H_{trans}$), 4.45 (d, *J* = 3.5 Hz, $1H_{cis}$), 3.82 (d, *J* = 3.5 Hz, $1H_{cis}$), 3.61 (d, *J* = 1.8 Hz, $1H_{trans}$).**^{13}C NMR** (75 MHz, $CDCl_3$) δ 135.86 (*trans*), 134.20 (*cis*), 128.26 (*trans*), 127.75 (*cis*), 127.61 (*trans*), 127.45(*cis*), 127.10 (*cis*), 127.03 (*trans*), 115.63 (*trans*), 115.12 (*cis*), 55.59 (*trans*), 54.89 (*cis*), 45.82 (*cis*), 45.43(*trans*).**IR** (cm^{-1})ν_{max}: 3110, 3029, 2247, 1444, 1404, 1308, 1246, 1225, 883, 714.**HRMS** (ESI, TOF MS) m/z calculated for $[M+H]^+$: 152.0170, found: 152.0172.



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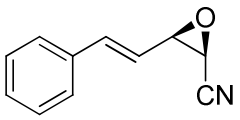


9'

(E)-3-Styryl-oxirane-2-carbonitrile

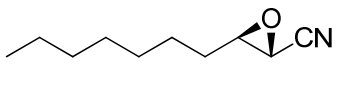
Epoxides **9** and **9'** were synthesized following general procedure starting from 4 g of cinnamaldehydeand a mixture of *cis*/*trans*stereoisomers 1:1.8 was isolated asacolorlessoil after rapid purification on alumina(1.6 g, 31% yield).(EtOAc/PE: 1 / 9, Rf_{trans} = 0.51, Rf_{cis} = 0.47). Additional analytical samples of pure isomers were obtained in low yield during this

purification (this compound is not stable upon attempted purification on silica gel chromatography).

Trans stereoisomer: **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.26 (m, 5H), 6.81 (d, *J* = 16.0 Hz, 1H), 5.67 (dd, *J* = 16.0, 7.8 Hz, 1H), 3.83 (dd, *J* = 7.8, 1.7 Hz, 1H), 3.32 (d, *J* = 1.8 Hz, 1H).**^{13}C NMR** (75 MHz, $CDCl_3$) δ 138.21, 134.98, 129.13, 128.89, 126.84, 121.49, 116.20, 58.94, 43.31.

*Cis*stereoisomer :**^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.33 (m, 5H), 6.92 (d, *J* = 15.9 Hz, 1H), 5.98 (dd, *J* = 15.9, 8.1 Hz, 1H), 3.76 (dd, *J* = 8.1, 3.7 Hz, 1H), 3.65 (d, *J* = 3.7 Hz, 1H).**^{13}C NMR** (75 MHz, $CDCl_3$) δ 139.42, 135.04, 129.12, 128.82, 126.97, 120.30, 115.54, 57.54, 43.64.

IR (cm^{-1}) ν_{max}: 2245, 1490, 1451, 1412, 1226, 974, 891, 773, 695.**HRMS** (ESI, TOF MS) m/z calculated for $[M+H]^+$: 172.0762, found: 172.0763.



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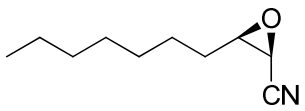


11'

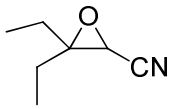
3-Heptyl-oxirane-2-carbonitrile

To a solution of octanal (2g, 12 mmol),chloroacetonitrile (1.3 g, 17 mmol) and a catalytic amount (50 mg) of dodecyltrimethylammonium chloride (DTA-Cl)inacetonitrile (30 mL) was added under stirring a solution of 50% aqueous NaOH (30 mL).After 20 minutes, full conversion of the aldehyde was evidenced by TLC and the reaction mixture was diluted with water (50 mL) and DCM (50 mL). The aqueous layer was extracted with dichloromethane and washed with brine, concentrated under reduced pressure and purifiedon a short plug of silica gel using PE/EtOAc 9:1 as eluant.A 1:1 mixture of **9** and **9'** was isolated as a colorless liquid (1.5 g, 59% yield). (EtOAc/PE: 1/9, Rf_{trans} = 0.67, Rf_{cis} = 0.65). Further purification by silica gel chromatography using PE/Et_2O 9:1 allowed to isolate pure*trans* and *cis*isomers.

Trans stereoisomer:**^{1}H NMR** (300 MHz, $CDCl_3$) δ 3.34 (td, *J* = 6.0, 1.9 Hz, 1H), 3.17 (d, *J* = 1.9 Hz, 1H), 1.71–1.20 (m, 12H), 0.89 (t, *J* = 6.5 Hz, 3H).**^{13}C NMR** (75 MHz, $CDCl_3$) δ 116.80, 59.17, 40.95, 31.63, 31.01, 29.08, 29.02, 25.26, 22.57, 14.04.

Cis stereoisomer: **^{1}H NMR** (300 MHz, $CDCl_3$) δ 3.46 (d, *J* = 3.8 Hz, 1H), 3.19 (td, *J* = 6.0, 3.7 Hz, 1H), 1.88 – 1.17 (m, 12H), 0.90 (t, *J* = 6.6 Hz, 3H).**^{13}C NMR** (75 MHz, $CDCl_3$) δ 115.94, 57.44, 41.74, 31.66, 29.75, 29.15, 29.06, 25.72, 22.59, 14.07.

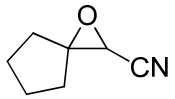
HRMS (ESI, TOF MS) m/z calculated for $[M+H]^+$: 168.1388, found: 168.1387.



12

3,3-Diethyl-oxirane-2-carbonitrile

Epoxide **12**was synthesized following general procedure starting from 3 g of 3-pentanone (34.8 mmol)and was isolated as a colorlessoil(2.4 g, 55% yield).(EtOAc/PE: 1/9, Rf = 0.75). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 3.27 (s, 1H), 1.96–1.62 (m, 4H), 1.09 (td, *J* = 7.5, 1.3 Hz, 3H), 0.92 (td, *J* = 7.5, 1.3 Hz, 3H).**^{13}C NMR** (75 MHz, $CDCl_3$) δ 116.48, 67.34, 46.21, 25.44, 25.12, 9.07, 8.31.**IR** (cm^{-1})ν_{max}: 2976, 2944, 2884, 2244, 1462, 1411, 938, 909, 820.**HRMS** (ESI, TOF MS) m/z calculated for $[M+H]^+$: 126.0919, found: 126.0917.



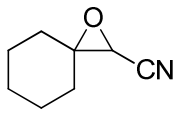
13

1-Oxa-spiro[2.4]heptane-2-carbonitrile

Epoxide **13**was synthesized following general procedure starting from 2 g of cyclopentanone(24mmol) and was isolated as a colorlessoil(2.4 g, 82% yield). (EtOAc/PE: 1/9, Rf = 0.57).

^{1}H NMR (300 MHz, $CDCl_3$) δ 3.46 (s, 1H), 2.17–1.60 (m, 8H).**^{13}C NMR** (75 MHz, $CDCl_3$) δ 116.59, 71.43, 45.78, 31.84, 30.62, 25.33, 24.92.**IR** (cm^{-1}) ν_{max}: 2967, 2875, 2244, 1453,

1436, 1413, 941.**HRMS** (ESI, TOF MS) m/z calculated for $[M+H]^+$: 124.0762, found: 124.0766.

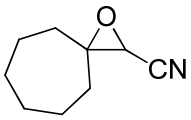


14

1-Oxa-spiro[2.5]octane-2-carbonitrile

Epoxide**14** was synthesized following general procedure starting from 2 g of cyclohexanone (20 mmol)and was isolated as acolorlessoil(2.4 g, 86% yield). (EtOAc/PE: 1/9, Rf = 0.64).

^{1}H NMR (300 MHz, $CDCl_3$) δ 3.24 (s, 1H), 1.83-1.49 (m, 10H).**^{13}C NMR** (75 MHz, $CDCl_3$) δ 116.27, 65.46, 47.39, 33.17, 31.07, 24.75, 24.58.**IR** (cm^{-1}) ν_{max}: 2968, 2244, 1413, 942. **HRMS** (ESI, TOF MS) m/z calculated for $[M+H]^+$: 138.0919, found: 138.0913

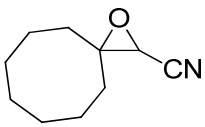


15

1-Oxa-spiro[2.6]nonane-2-carbonitrile

Epoxide**15** was synthesized following general procedure starting from 2 g of cycloheptanone(18 mmol) and was isolated as a colorless liquid(2.3 g, 85 % yield).(EtOAc/PE: 1/9, Rf = 0.60).

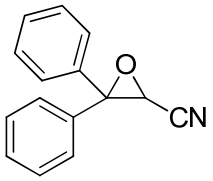
^{1}H NMR (300 MHz, $CDCl_3$) δ 3.25 (s, 1H), 2.12–1.44 (m, 12H).**^{13}C NMR** (75 MHz, $CDCl_3$) δ 116.46, 66.79, 48.58, 35.46, 33.17, 28.84, 28.68, 24.33, 23.99.**IR** (cm^{-1}) ν_{max}: 2930, 2859, 2244, 1468, 1448, 943. **HRMS** (ESI, TOF MS) m/z calculated for $[M+H]^+$: 152.1075, found: 152.1071.



16

1-Oxa-spiro[2.7]decane-2-carbonitrile

Epoxide**16** was synthesized following general procedure starting from 2 g of cyclooctanone (16mmol)and was isolated as a yellow liquid(1.3 g, 48% yield).(EtOAc/PE: 1/9, Rf = 0.60). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 3.28 (s, 1H), 2.01–1.46 (m, 14H).**^{13}C NMR** (75 MHz, $CDCl_3$) δ 116.50, 66.29, 49.11, 33.76, 31.74, 26.45, 25.89, 24.96, 24.35, 22.69.**IR**(cm^{-1}) ν_{max}: 2933, 2859, 2243, 1456, 1252, 967, 939. **HRMS** (ESI, TOF MS) m/z calculated for $[M+H]^+$: 166.1232, found: 166.1233.

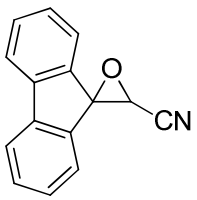


17

3,3-Diphenyl-oxirane-2-carbonitrile

Epoxide**17** was synthesized following general procedure starting from 3 g of benzophenoneand was isolated as a white solid(3.2 g, 88% yield). (EtOAc/PE: 1/9, Rf = 0.40).

Mp: 75°C.**^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.41–7.21 (m, 10H), 3.83 (s, 1H).**^{13}C NMR** (75 MHz, $CDCl_3$) δ 136.44, 134.48, 129.34, 129.32, 128.81, 128.65, 127.72, 127.34, 115.27, 67.68, 50.11.**IR** (cm^{-1}) ν_{max}: 3061, 3026, 2248, 1495, 1445, 1297, 1268, 935, 916, 901, 766, 755, 703, 693, 651, 599. **HRMS** (ESI, TOF MS) m/z calculated for $[M+H]^+$: 222.0919, found: 222.0919.



18

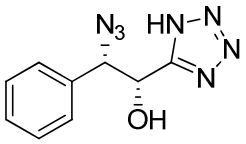
3,3-Fluoren-9-yl-oxirane-2-carbonitrile

Epoxide **18**was synthesized following general procedure starting from 3 g of fluorenoneand was isolated as awhite solid(1.53 g, 42% yield). (EtOAc/PE : 1/9, Rf = 0.45).

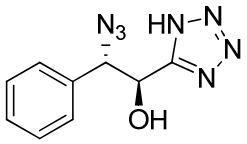
Mp:122°C.**^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.65–7.02 (m, 8H), 4.12 (s, 1H).**^{13}C NMR** (75 MHz, $CDCl_3$) δ 141.57, 141.35, 137.30, 135.48, 130.86, 130.78, 128.15, 127.99, 123.78, 121.55, 120.82, 120.78, 115.17, 67.20, 48.31.**IR** (cm^{-1}) ν_{max}: 3070, 2247, 1615, 1491, 1398, 1325, 1295, 916, 759, 747, 725. **HRMS** (ESI, TOF MS) m/z calculated for $[M+H]^+$: 220.0762, found: 220.0759.

General procedure for the synthesis of α-hydroxy-β-azido tetrazoles:

A solution ofcyanoepoxide (1equiv.), Bu_2SnO (0.5 equiv.) and $TMSN_3$(3 equiv.) in toluene (10 mL/mmol of epoxide) was stirred at 60°C for 18 h. The solvent was removed under reduced pressure anda 1:1 THF/2N aqueousHClmixture (20 mL) was added to the crude and stirred for 30 min. Water and EtOAc were added and the aqueous layer was extracted (EtOAc), washed with brine and dried over $MgSO_4$. Evaporation gave a residue that was washed by trituration with small portions of dichloromethane.Further purification could be done by flash chromatography over silica gel using dichloromethane/ MeOH / Acetic acid : 9 / 0.5 / 0.5 mixture as eluent (spots were revealed with ninhydrin).



2



19

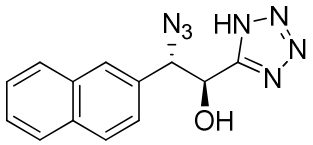
Syn and Anti-2-azido-2-phenyl-1-(1H-tetrazol-5-yl)ethanol

Tetrazoles**2** and **19** were synthesized following general procedure starting from 183 and148 mgof cyanoepoxide**1** and **3** respectively. Products were isolated as white solids.(**2**: 190 mg, 65% yield; **19**: 200 mg, 85% yield). (Dichloromethane/ MeOH / Acetic acid : 9/0.5/0.5, Rf = 0.38).**Mp**: **2**:176°C, **19**: 175°C.

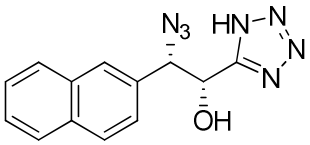
Anti stereoisomer: **^{1}H NMR** (300 MHz, DMSO) δ7,40-7,20 (m, 5H), 6,76 (dl, J=4,9 Hz, 1H), 5,33 (t, J=5,3 Hz, 1H), 5,16 (d, J=6,0 Hz, 1H).**^{13}C NMR** (75 MHz, DMSO) δ156,76, 135,34, 128,31, 127,85, 67,58, 67,39.

Syn-stereoismer: **^{1}H NMR** (300 MHz, DMSO) δ7,35-7,20 (m, 5H), 6,88 (dl, J=5,8 Hz, 1H), 5,28 (t, J=5,6 Hz, 1H), 5,07 (d, 1H, J=6,1 Hz).**^{13}C NMR** (75 MHz, DMSO) δ135,71, 128,49, 127,89, 68,13, 68,05.

IR (cm^{-1}) ν_{max}: 3370, 2628, 2113, 1076, 753, 696, 594, 554. **HRMS** (ESI, TOF MS) m/z calculated for $[M+H]^+$: 232,0947, found: 232,0950.



20



21

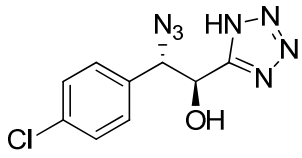
Syn and Anti 2-azido-2-(naphthalen-2-yl)-1-(1H-tetrazol-5-yl)ethanol

Tetrazoles**20** and **21** were synthesized following general procedure starting from 150 mg of cyanoepoxide**4** and **5** respectively. Products were isolated as white solids. (**20**: 216mg, 55% yield,**21**: 121mg, 56% yield). (Dichloromethane/ MeOH / Acetic acid : 9/0.5/0.5, Rf = 0.35).**Mp**: **20**: 211°C (dec.),**21**: 209°C (dec.).

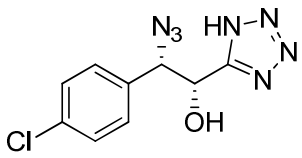
Anti stereoisomer: **^{1}H NMR** (300 MHz, DMSO) δ8,00-7,85 (m, 3H), 7,82 (s, 1H), 7,60-7,45 (m, 2H), 7,42 (d, 1H, J=8,6 Hz), 6,83 (dl, J=4,7 Hz, 1H), 5,45 (t, J=5,0 Hz, 1H), 5,36 (d, J=5,9 Hz, 1H).**^{13}C NMR** (75 MHz, DMSO) δ132,95, 132,66, 132,44, 127,92, 127,50, 127,17, 126,42, 126,36, 125,31, 67,78, 67,47.

Syn-stereoismer: **^{1}H NMR** (300 MHz, DMSO) δ7,90-7,80 (m, 4H), 7,60-7,40 (m, 4H), 5,41 (d, J=6,4 Hz, 1H), 5,36 (d, J=6,4 Hz, 1H).**^{13}C NMR** (75 MHz, DMSO) δ133,26,64, 132,44, 128,02, 127,91, 127,51, 127,05, 126,46, 126,39, 125,35, 68,35, 68,11.

IR (cm^{-1}) ν_{max}: 3338, 2357, 2105, 1245, 1078, 817, 746, 555, 477. **HRMS** (ESI, TOF MS) m/z calculated for $[M+H]^+$: 282.1103, found: 282.1101



22



23

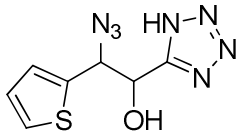
Syn and Anti2-azido-2-(4-chlorophenyl)-1-(1H-tetrazol-5-yl)ethanol

Tetrazoles**22** and **23** were synthesized following general procedure starting from 160 mg of cyanoepoxide**6** and **7** respectively. Products were isolated as white solids.(**22**: 150mg, 63% yield,**23**: 121mg, 51% yield). (Dichloromethane/ MeOH / Acetic acid: 9/0.5/0.5, Rf = 0.33).Mp: **22**: 198°C (dec.) **23**:203°C (dec.).

Anti stereoisomer: **^{1}H NMR** (300 MHz, DMSO) δ7,40 (d, J=8,5 Hz, 2H), 7,28 (d, J=8,5 Hz, 2H), 6,82 (dl, J=3,2 Hz, 1H), 5,34 (t, J=4,9 Hz, 1H), 5,22 (d, J=5,7 Hz, 1H).**^{13}C NMR** (75 MHz, DMSO) δ134,36, 132,93, 129,72, 128,27, 67,43, 66,75.

Syn-stereoismer: **^{1}H NMR** (300 MHz, DMSO) δ7,42 (d, J=8,6 Hz, 2H), 7,34 (d, J=8,6 Hz, 2H), 6,91 (dl, J=5,5 Hz, 1H), 5,29 (t, J=5,3 Hz, 1H), 5,14 (d, J=6,0 Hz, 1H).**^{13}C NMR** (75 MHz, DMSO) δ134,73, 133,03, 129,74, 128,39, 67,93, 67,18.

IR (cm^{-1}) ν_{max}: 3373, 2116, 1492, 1244, 1073, 875, 824, 797, 600, 522, 480. **HRMS** (ESI, TOF MS) m/z calculated for $[M+H]^+$: 266.0560, found: 266.0557.

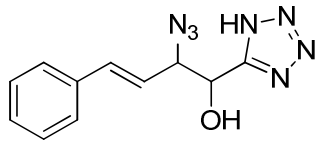


24

Syn and anti-2-azido-1-(1H-tetrazol-5-yl)-2-thiophen-2-yl-ethanol

Tetrazole**24** was synthesized following general procedure starting from 1g of cyanoepoxide**8** (1:2 mixture of *cis* / *trans* stereoisomers).Product was purified by flash chromatography using dichloromethane/ MeOH / acetic acid :15 / 0.5 / 0.5mixture as eluantand was isolated as

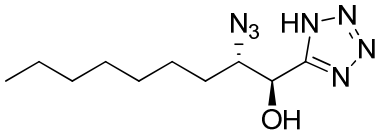
brown solid (590 mg, 38% yield,1:1.3 mixture of two stereoisomers).(Dichloromethane/ MeOH / acetic acid : 9 / 0.5 / 0.5, Rf = 0.36).**Mp**: 125°C.**^{1}H NMR** (300 MHz, DMSO) δ 7.57 (d, *J* = 5.1 Hz, 1H$_{min}$), 7.51 (d, *J* = 5.1 Hz, 1H$_{maj}$), 7.19 (d, *J* = 2.8 Hz, 1H$_{min}$), 7.12 (d, *J* = 2.8 Hz, 1H$_{maj}$), 7.07 (s, 1H), 7.03 (dd, *J* = 5.1, 3.6 Hz, 1H$_{min}$), 6.99 (dd, *J* = 5.0, 3.6 Hz, 1H$_{maj}$), 5.45 (d, *J* = 4.8 Hz, 1H$_{maj}$), 5.36 (d, *J* = 4.5 Hz, 1H$_{maj}$ + 1H$_{min}$), 5.32 (d, *J*= 4.3 Hz, 1H$_{min}$). **^{13}C NMR** (75 MHz, DMSO) δ 156.94 (*maj*), 156.21 (*min*), 137.18 (*min*), 136.35 (*maj*), 128.00 (*maj*), 127.84 (*min*), 127.34 (*min*), 127.23 (*maj*), 126.51 (*min*), 67.93 (*min*), 67.54 (*maj*), 63.35 (*min*), 63.32 (*maj*).**IR** (cm^{-1}) ν$_{max}$: 3370, 3105, 2719, 2114, 1317, 1282, 1226, 1077, 714. **HRMS** (ESI, TOF MS) m/z calculated for [M+H]$^{+}$: 238.0511, found: 238.0514.



25

Syn and anti-(E)-2-azido-4-phenyl-1-(1H-tetrazol-5-yl)-but-3-en-1-ol

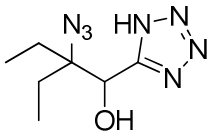
Tetrazole**25** was synthesized following general procedure starting from 500 mg ofstereoisomerically pure *trans*-cyanoepoxide**9**and was isolated as a white solid (390 mg, 52% yield). Mixture of two stereoisomers (2:1 ratio).(Dichloromethane/MeOH/Acetic acid: 9/0.5/0.5, Rf = 0.31).Mp: 134°C.**^{1}H NMR** (300 MHz, DMSO) δ 7.52-7.23 (m, 5H),6.85 (s, 1H), 6.81 (d, *J* = 16.0 Hz, 1H$_{min}$), 6.69 (d, *J* = 15.9 Hz, 1H$_{maj}$), 6.38 (dd, *J* = 15.9, 8.0 Hz, 1H$_{min}$), 6.31 (dd, *J* = 15.9, 8.0 Hz, 1H$_{maj}$), 5.25 (bm, 1H$_{min}$), 5.24 (bm, 1H$_{maj}$), 4.66 (dd, *J* = 7.5, 4.5 Hz, 1H$_{maj}$), 4.58 (dd, *J* = 7.8, 4.4 Hz, 1H$_{min}$).**^{13}C NMR** (75 MHz, DMSO) δ 156.33, 135.55 (*min*), 135.52 (*maj*), 135.07 (*maj*), 134.89 (*min*), 128.69 (*maj*), 128.29 (*min*), 126.63 (*min*), 126.58 (*maj*), 122.79 (*min*), 122.27 (*maj*), 67.34 (*min*), 67.08 (*maj*), 66.68 (*maj*), 66.51(*min*).**IR** (cm^{-1}) ν$_{max}$: 3356, 3025, 2718, 2106, 1576, 1448, 1246, 1114, 965, 752, 691. **HRMS** (ESI, TOF MS) m/z calculated for [M+H]$^{+}$: 258.1103, found: 258.1102.



26

Anti-2-azido-1-(1H-tetrazol-5-yl)-nonan-1-ol

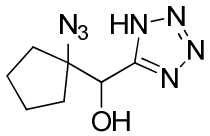
Tetrazole**26** was synthesized following general procedure starting from 410 mg of *trans*-cyanoepoxide**11**.Instead of precipitation, crude product was purified by flash chromatography using dichloromethane/ MeOH / Acetic acid: 9 / 0.5 / 0.5 mixture as eluant. Product was isolated as white solid (440 mg, 70% yield). (Dichloromethane/ MeOH / Acetic acid: 9/0.5/0.5, Rf = 0.37).**Mp**: 146°C.**^{1}H NMR** (300 MHz, DMSO) δ 6.69 (s, 1H), 5.15 (d, *J* = 3.9 Hz, 1H), 3.85–3.75 (m, 1H), 1.60–1.10 (m, 12H), 0.89–0.79 (m, 3H).**^{13}C NMR** (75 MHz, DMSO) δ 156.27, 67.31, 65.57, 31.10, 29.01, 28.56, 28.45, 25.51, 22.02, 13.88.**IR** (cm^{-1}) ν_{max}: 3377, 2933, 2858, 2661, 2101, 1571, 1463, 1439, 1250, 1113, 1093, 1064, 940. **HRMS** (ESI, TOF MS) m/z calculated for $[M+H]^+$: 254.1729, found: 254.1732.



27

2-Azido-2-ethyl-1-(1H-tetrazol-5-yl)-butan-1-ol

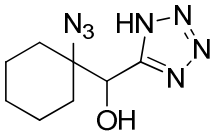
Tetrazole**27** was synthesized following general procedure starting from 1g of cyanoepoxide**12**and was isolated as a white solid (1 g, 60% yield). (Dichloromethane/MeOH/Acetic acid: 9/0.5/0.5, Rf = 0.36).**Mp**: 128°C.**^{1}H NMR** (300 MHz, DMSO) δ 6.74 (d, *J* = 4.3 Hz, 1H), 5.10 (d, *J* = 4.2 Hz, 1H), 1.95-1.75 (m, 2H), 1.45-1.25 (m, 2H), 0.94 (t, *J* = 7.3 Hz, 3H), 0.80 (t, *J* = 7.3 Hz, 3H).**^{13}C NMR** (75 MHz, DMSO) δ 156.27, 68.60, 24.24, 23.04, 7.55, 7.36.**IR** (cm^{-1}) ν_{max}: 3393, 2978, 2948, 2596, 2112, 1455, 1262, 1117, 1081, 1058, 921. **HRMS** (ESI, TOF MS) m/z calculated for $[M+H]^+$: 212.1260, found: 212.1262.



28

(1-Azido-cyclopentyl)-(1H-tetrazol-5-yl)-methanol

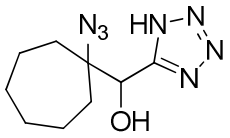
Tetrazole**28** was synthesized following general procedure starting from 1 g of cyanoepoxide and was isolated as awhite solid(900 mg, 53% yield). (Dichloromethane/MeOH/Acetic acid : 9/0.5/0.5, Rf = 0.41).**Mp**: 161°C.**^{1}H NMR** (300 MHz, DMSO) δ 6.78 (d, *J* = 4.5 Hz, 1H), 5.10 (d, *J* = 4.4 Hz, 1H), 1.97-1.75 (m, 2H), 1.45-1.22 (m, 6H). **^{13}C NMR** (75 MHz, DMSO) δ 156.68, 75.15, 70.60, 33.55, 33.11, 23.41, 23.30.**IR** (cm^{-1}) ν_{max}: 3377, 2973, 2958, 2874, 2595, 2117, 1452, 1250, 1071, 948. HRMS (ESI, TOF MS) m/z calculated for $[M+H]^+$: 210.1103, found: 210.1104



29

(1-Azido-cyclohexyl)-(1H-tetrazol-5-yl)-methanol

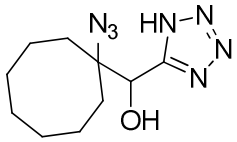
Tetrazole**29** was synthesized following general procedure starting from 500 mg of cyanoepoxide**14**and was isolated as a white solid(610 mg, 75% yield). (Dichloromethane/MeOH/Acetic acid: 9/0.5/0.5, Rf = 0.47).**Mp**: 175°C.**^{1}H NMR** (300 MHz, DMSO) δ 6.80 (d, *J* = 4.8 Hz, 1H), 5.06 (d, *J* = 4.8 Hz, 1H), 2.23–0.91 (m, 10H). **^{13}C NMR** (75 MHz, DMSO) δ 156.01, 71.40, 65.48, 30.23, 29.77, 24.54, 21.39, 21.14.**IR** (cm^{-1}) ν_{max}: 3369, 2938, 2857, 2582, 2102, 1449, 1255, 1120, 1065, 952. **HRMS** (ESI, TOF MS) m/z calculated for $[M+H]^+$: 224.1260, found: 224.1264.



30

(1-Azido-cycloheptyl)-(1H-tetrazol-5-yl)-methanol

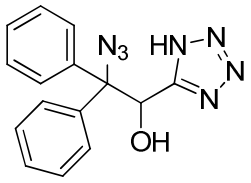
Tetrazole**30** was synthesized following general procedure starting from 1 g of cyanoepoxide**15**and was isolated as awhite solid(1.2 g, 76% yield). (Dichloromethane/MeOH/Acetic acid: 9/0.5/0.5, Rf = 0.45).**Mp**: 172°C.**^{1}H NMR** (300 MHz, DMSO) δ 6.81 (d, *J* = 4.8 Hz, 1H), 5.00 (d, *J* = 4.8 Hz, 1H), 2.19–1.04 (m, 12H). **^{13}C NMR** (75 MHz, DMSO) δ 156.21, 70.95, 68.96, 33.67, 33.07, 28.91, 28.88, 21.75, 21.66.**IR** (cm^{-1}) ν_{max}: 3348, 2932, 2856, 2105, 1458, 1436, 1257, 1066, 942.**HRMS** (ESI, TOF MS) m/z calculated for $[M+H]^+$: 238.1416, found: 238.1408.



31

(1-Azido-cyclooctyl)-(1H-tetrazol-5-yl)-methanol

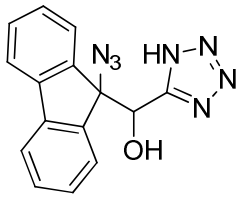
Tetrazole**31** was synthesized following general procedure starting from 600 mg of cyanoepoxide**16** and was isolated as awhite solid(470mg, 51% yield). (Dichloromethane/MeOH/Acetic acid: 9/0.5/0.5, Rf = 0.44).**Mp**: 186°C.**^{1}H NMR** (300 MHz, DMSO) δ 6.76 (d, *J* = 4.6 Hz, 1H), 5.06 (d, *J* = 4.6 Hz, 1H), 2.23–1.88 (m, 2H), 1.77–1.18 (m, 12H). **^{13}C NMR** (75 MHz, DMSO) δ 156.26, 70.09, 68.91, 29.79, 28.06, 27.71, 27.21, 24.10, 21.49, 21.26.**IR**(cm^{-1}) ν_{max}: 3333, 2931, 2588, 2104, 1742, 1447, 1253, 1083, 1058. **HRMS** (ESI, TOF MS) m/z calculated for $[M+H]^+$: 252.1573, found: 252.1576.



32

2-Azido-2,2-diphenyl-1-(1H-tetrazol-5-yl)-ethanol

Tetrazole**32** was synthesized following general procedure starting from 2g of cyanoepoxide**17** and was isolated as white solid(1.7 g, 61% yield).(Dichloromethane/MeOH/Acetic acid: 9/0.5/0.5, Rf = 0.21).**Mp**: 205°C.**^{1}H NMR** (300 MHz, DMSO) δ 7.63–7.29 (m, 5H), 7.22–6.99 (m, 6H), 6.39 (d, *J* = 4.2 Hz, 1H). **^{13}C NMR** (75 MHz, DMSO) δ 155.27, 140.06, 128.56, 127.95, 127.93, 127.71, 127.25, 126.11, 74.31, 69.88.**IR** (cm^{-1}) ν_{max}: 3333, 2110, 1446, 1268, 1100, 1062, 744, 720, 699, 592. **HRMS** (ESI, TOF MS) m/z calculated for $[M+H]^+$: 308.1260, found: 308.1252.



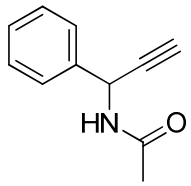
33

(9-Azido-9H-fluoren-9-yl)-(1H-tetrazol-5-yl)-methanol

Tetrazole**33** was synthesized following general procedure starting from 2g of cyanoepoxide**18**and was isolated as white solid(1.7 g, 61% yield). (Dichloromethane/MeOH/Acetic acid: 9/0.5/0.5, Rf = 0.21).Mp: 213°C.**^{1}H NMR** (300 MHz, DMSO) δ 7.82 (d, *J* = 6.9 Hz, 2H), 7.57–7.35 (m, 6H), 7.31 (d, *J* = 5.0 Hz, 1H), 5.68 (d, *J* = 4.8 Hz, 1H). **^{13}C NMR** (75 MHz, DMSO) δ 155.35, 141.47, 140.69, 140.13, 139.92, 129.87, 127.94, 127.81, 125.47, 125.14, 120.44, 74.21, 69.45.**IR** (cm^{-1}) ν_{max}: 3393, 2646, 2110, 1451, 1250, 1112, 1085, 744, 733. **HRMS** (ESI, TOF MS) m/z calculated for $[M+H]^+$: 306.1103, found: 306.1111.

General procedure for the reaction of α-Hydroxy-β-Azido Tetrazoles with triphenylphosphine

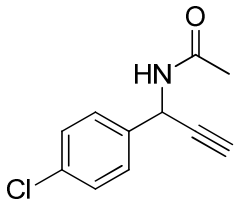
A solution of tetrazole and triphenylphosphine (1.2 equiv.) in THF (20 mL/mmol) was stirred at reflux for 2h. The reaction mixture was cooled down to room temperature and triethylamine (3 equiv.) followed by acetic anhydride (3 equiv.) were added and the mixture was stirred at room temperature for 1h. Usual workup (water and EtOAc) gave a residue that was purified byflash chromatography on silica gel.



35

N-(1-Phenyl-prop-2-ynyl)-acetamide

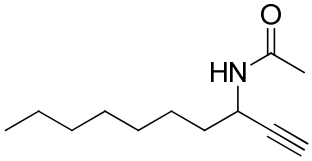
Acetamide**35** was synthesized following the general procedure starting from 100 mg of **2**and purified byflash chromatography using PE/EtOAc7:3 as eluant. The product was isolated as a white solid (39 mg, 52% yield). **Mp**: 80°C.**^{1}H NMR** (200 MHz, $CDCl_3$) δ 7.65–7.30 (m, 5H), 6.36 (d, *J* = 7.9 Hz, 1H), 6.01 (dd, *J* = 8.5, 2.4 Hz, 1H), 2.49 (d, *J* = 2.4 Hz, 1H), 2.00 (s, 3H).**^{13}C NMR** (75 MHz, $CDCl_3$) δ 167.98, 137.24, 127.71, 127.21, 126.01, 80.72, 71.93, 43.44, 22.06.**IR** (cm^{-1}) ν_{max}:3291, 3063, 2929, 1651, 1538, 1493, 1446, 1370, 1307, 1262, 1098, 1076, 756, 742, 696, 652, 596, 561. **HRMS** (ESI, TOF MS) m/z calculated for $[M+H]^+$: 306.1103, found: 306.1111.



36

N-(1-(4-chlorophenyl)prop-2-yn-1-yl)acetamide

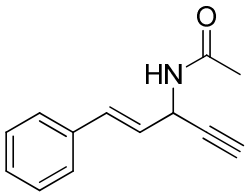
Acetamide**36** was synthesized following the general procedure starting from 102 mg of **23** and purified by flash chromatography using PE/EtOAc7:3 as eluant.The product was isolated as a white solid (57 mg, 71% yield). **Mp**: 114°C.**^{1}H NMR** (200 MHz, $CDCl_3$)δ 7.50-7.40 (m, 2H), 7.37-7.28 (m, 2H), 6.25 (d, *J* = 8.0 Hz, 1H), 5.98 (dd, *J* = 8.4, 2.4 Hz, 1H), 2.51 (d, *J* = 2.4 Hz, 1H), 2.02 (s, 3H).^{13}C NMR (75 MHz, $CDCl_3$) δ169.03, 136.89, 134.13, 128.86, 128.47, 81.20, 73.45, 43.91, 23.11.**IR** (cm^{-1}) ν_{max}:3282, 1648, 1525, 1490, 1366, 1300, 1281, 1094, 1013, 657, 595, 559. **HRMS** (ESI, TOF MS) m/z calculated for $[M+H]^+$: 230.0349, found: 230.0346.



37

N-(dec-1-yn-3-yl)acetamide

Acetamide**37** was synthesized following the general procedure starting from 131 mg of **26** and purified by flash chromatography using PE/EtOAc7:3 as eluant. The product was isolated as an oil crystallizing on standing (30 mg, 30% yield). **Mp**: 57°C.**^{1}H NMR** (200 MHz, $CDCl_3$)δ5.70 (d, *J* = 8.2 Hz, 1H), 4.72(m, 1H), 2.25 (d, *J* = 2.4 Hz, 1H), 1.99 (s, 3H), 1.73-1.57 (m, 2H), 1.50-1.15 (m, 10H), 0.93-0.80 (m, 3H).^{13}C NMR (75 MHz, $CDCl_3$) δ 169.12, 136.89, 83.42, 71.16, 41.48, 35.85, 31.87, 29.27, 29.9, 25.67, 23.39, 22.75, 14.22.**IR** (cm^{-1}) ν_{max}:3284, 3251, 2951, 2852, 1634, 1542, 1370, 745, 675, 605, 544. **HRMS** (ESI, TOF MS) m/z calculated for $[M+H]^+$: 196.1701, found: 196.1700.

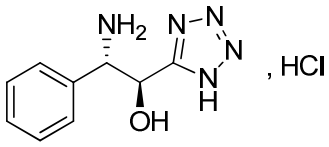


38

(E)-N-(1-phenylpent-1-en-4-yn-3-yl)acetamide

Acetamide**38** was synthesized following the general procedure starting from 73 mg of **25** and purified by flash chromatography using PE/EtOAc6:4 as eluant.The product was isolated as a white solid (13 mg, 23% yield). **Mp**: 119°C.**^{1}H NMR** (200 MHz, $CDCl_3$)δ7.45-7.25 (m, 5H), 6.85 (dd, *J* = 15.8, 1.5 Hz, 1H), 6.16 (dd, *J* = 15.8, 5.2 Hz, 1H) 6.00 (d, *J* = 8.0 Hz, 1H), 5.66-5.52 (m, 1H)2.50 (d, *J* = 2.2 Hz, 1H), 2.04 (s, 3H).**^{13}C NMR** (75 MHz, $CDCl_3$) δ 169.09, 135.99, 132.47, 128.75, 128.26, 126.82, 125.32, 80.88, 73.40, 42.56, 23.31.**IR** (cm^{-1}) ν_{max}:3290, 1642, 1535, 1446, 1370, 966, 749, 640, 545. **HRMS** (ESI, TOF MS) m/z calculated for $[M+H]^+$: 200.1075, found: 200.1076.

Procedure for the hydrogenation of 19



39

To a solution of tetrazole**19**(252 mg, 1.09 mmol) in a MeOH/HCl 0.5 N 5:3 mixture (20 mL) was added Pd/C 10% (100 mg). The reaction mixture was stirred overnight under hydrogen pressure (0.5 bar) and filtered on Celite using a MeOH/HCl 0.5 N 1:1 mixture as eluent. The solvent was then removed and product **39**collected as a white solid (264 mg, quant. yield).

Mp: 182°C. **^{1}H NMR** (200 MHz, MeOD) δ 7.32 (s, 5H), 5.68 (d, *J* = 8.0 Hz, 1H), 4.75 (d, *J*= 8.0 Hz, 1H).**^{13}C NMR** (75 MHz, MeOD) δ133.28, 130.6, 129.83, 129.63, 67.13,

59.31.**IR**(cm^{-1}) ν_{max}: 2790, 1588, 1503, 1454, 1135, 1059, 712, 702.**HRMS** (ESI, TOF MS) m/z calculated for $[M+H]^+$: 206.1042, found: 206.1045.

X-Ray Data for Compound 2

Crystal data

$C_9H_9N_7O$
$M_r = 231.23$
Orthorhombic, $Pca2_1$
$a = 9.9162$ (8) Å
$b = 14.8123$ (11) Å
$c = 7.3118$ (6) Å
$V = 1073.97$ (15) Å^3
$Z = 4$
$F(000) = 480$

$D_x = 1.430$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9531 reflections
$\theta = 2.8$–$30.0°$
$\mu = 0.10$ mm^{-1}
$T = 200$ K
Platelet, colorless
0.30 × 0.22 × 0.02 mm

Data collection

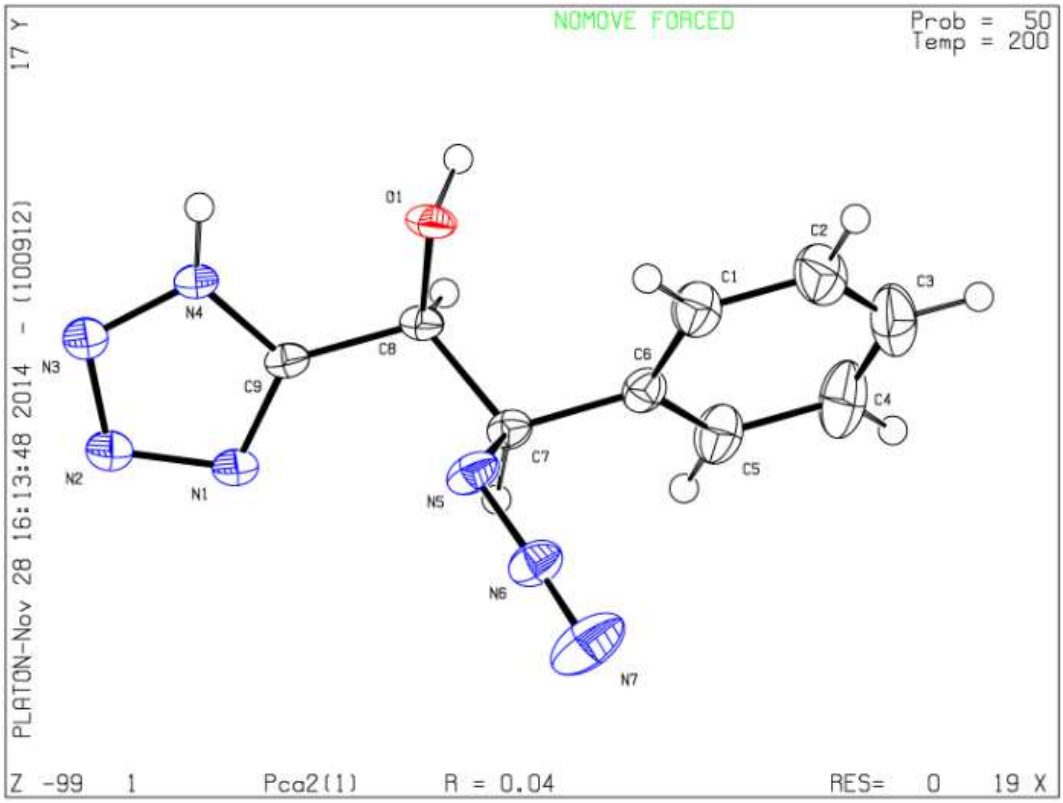
Bruker APEX-II CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan *SADABS* (Sheldrick, V2.10)
$T_{min} = 0.970$, $T_{max} = 0.998$

33251 measured reflections
1687 independent reflections
1462 reflections with $I > 2\sigma(I)$
$R_{int} = 0.055$
$\theta_{max} = 30.1°$, $\theta_{min} = 2.5°$
$h = -13 \rightarrow 13$
$k = -20 \rightarrow 20$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.087$
$S = 1.06$
1687 reflections

159 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map



Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.77909 (10)	0.33818 (9)	0.97153 (19)	0.0239 (3)
H1A	0.8271	0.3252	0.8806	0.036*
C1	0.65461 (19)	0.15180 (13)	0.9496 (3)	0.0317 (4)
H1	0.7152	0.1693	1.0438	0.038*
C2	0.6663 (2)	0.06717 (14)	0.8695 (4)	0.0413 (6)
H2	0.7358	0.0273	0.9082	0.050*
C3	0.5779 (3)	0.04042 (14)	0.7341 (4)	0.0464 (6)
H3	0.5856	−0.0180	0.6813	0.056*
C4	0.4786 (3)	0.09867 (15)	0.6759 (4)	0.0479 (6)
H4	0.4180	0.0805	0.5822	0.057*
C5	0.4666 (2)	0.18395 (13)	0.7538 (3)	0.0338 (4)
H5	0.3982	0.2239	0.7121	0.041*
C6	0.55379 (17)	0.21136 (12)	0.8919 (3)	0.0235 (4)
C7	0.53840 (16)	0.30450 (11)	0.9729 (2)	0.0202 (3)
H7	0.4503	0.3301	0.9312	0.024*
C8	0.65116 (15)	0.36817 (11)	0.9114 (2)	0.0187 (3)
H8	0.6516	0.3704	0.7747	0.022*
C9	0.63538 (14)	0.46257 (11)	0.9829 (2)	0.0184 (3)
N1	0.52530 (13)	0.51005 (10)	1.0133 (2)	0.0242 (3)
N2	0.56736 (14)	0.59316 (10)	1.0724 (3)	0.0284 (4)
N3	0.69765 (14)	0.59628 (10)	1.0788 (3)	0.0276 (3)
N4	0.74110 (14)	0.51482 (9)	1.0227 (2)	0.0214 (3)
H4A	0.824 (2)	0.5036 (16)	1.020 (4)	0.042 (7)*
N5	0.53920 (15)	0.30304 (12)	1.1780 (2)	0.0264 (3)
N6	0.44715 (16)	0.25587 (11)	1.2417 (3)	0.0284 (3)
N7	0.3683 (2)	0.21459 (16)	1.3123 (3)	0.0519 (6)

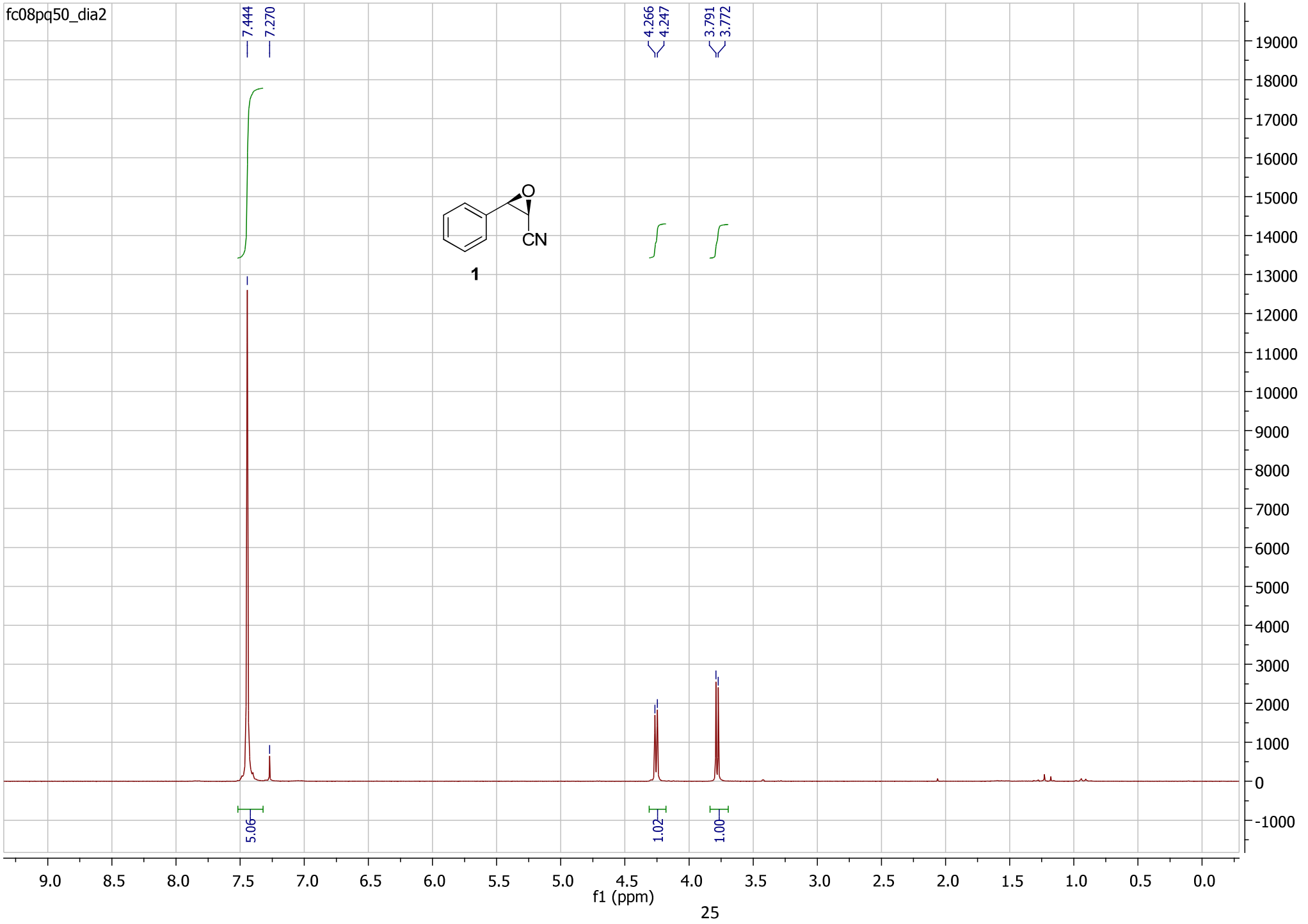
Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0136 (5)	0.0320 (6)	0.0262 (7)	0.0045 (4)	0.0017 (5)	−0.0028 (5)
C1	0.0354 (9)	0.0272 (9)	0.0324 (12)	0.0002 (7)	−0.0031 (9)	0.0049 (8)
C2	0.0507 (13)	0.0252 (10)	0.0479 (14)	0.0041 (9)	0.0080 (11)	0.0091 (10)
C3	0.0727 (16)	0.0249 (10)	0.0416 (13)	−0.0058 (10)	0.0103 (13)	−0.0023 (10)
C4	0.0683 (16)	0.0359 (12)	0.0395 (13)	−0.0145 (11)	−0.0112 (13)	−0.0078 (11)
C5	0.0372 (10)	0.0314 (10)	0.0327 (10)	−0.0053 (8)	−0.0089 (9)	−0.0008 (9)
C6	0.0247 (8)	0.0238 (8)	0.0219 (9)	−0.0041 (6)	0.0016 (7)	0.0018 (7)
C7	0.0171 (7)	0.0259 (8)	0.0175 (8)	−0.0015 (6)	−0.0018 (6)	−0.0009 (7)

C8	0.0143 (6)	0.0240 (8)	0.0179 (8)	0.0015 (6)	0.0002 (6)	−0.0003 (7)
C9	0.0141 (6)	0.0249 (8)	0.0163 (8)	−0.0011 (5)	0.0000 (6)	0.0007 (6)
N1	0.0153 (6)	0.0266 (7)	0.0309 (9)	0.0005 (5)	0.0012 (6)	−0.0023 (7)
N2	0.0187 (6)	0.0283 (7)	0.0382 (9)	0.0015 (5)	0.0017 (7)	−0.0049 (7)
N3	0.0195 (6)	0.0262 (7)	0.0370 (9)	0.0002 (5)	0.0001 (7)	−0.0049 (7)
N4	0.0129 (6)	0.0241 (6)	0.0272 (8)	0.0007 (5)	0.0006 (6)	−0.0011 (6)
N5	0.0225 (7)	0.0382 (9)	0.0186 (7)	−0.0101 (6)	0.0019 (6)	−0.0009 (7)
N6	0.0247 (7)	0.0381 (8)	0.0223 (8)	−0.0069 (6)	−0.0007 (6)	0.0010 (7)
N7	0.0445 (11)	0.0704 (14)	0.0407 (12)	−0.0263 (10)	0.0018 (9)	0.0121 (11)

Geometric parameters (Å, °)

O1—C8	1.4143 (18)	C7—N5	1.499 (2)
O1—H1A	0.8400	C7—C8	1.530 (2)
C1—C2	1.389 (3)	C7—H7	1.0000
C1—C6	1.398 (3)	C8—C9	1.501 (2)
C1—H1	0.9500	C8—H8	1.0000
C2—C3	1.380 (4)	C9—N1	1.317 (2)
C2—H2	0.9500	C9—N4	1.335 (2)
C3—C4	1.377 (4)	N1—N2	1.370 (2)
C3—H3	0.9500	N2—N3	1.294 (2)
C4—C5	1.391 (3)	N3—N4	1.345 (2)
C4—H4	0.9500	N4—H4A	0.84 (2)
C5—C6	1.390 (3)	N5—N6	1.240 (2)
C5—H5	0.9500	N6—N7	1.119 (2)
C6—C7	1.509 (3)		
C8—O1—H1A	109.5	C6—C7—C8	111.96 (14)
C2—C1—C6	120.14 (19)	N5—C7—H7	108.4
C2—C1—H1	119.9	C6—C7—H7	108.4
C6—C1—H1	119.9	C8—C7—H7	108.4
C3—C2—C1	120.6 (2)	O1—C8—C9	106.12 (13)
C3—C2—H2	119.7	O1—C8—C7	111.73 (13)
C1—C2—H2	119.7	C9—C8—C7	113.28 (13)
C4—C3—C2	119.7 (2)	O1—C8—H8	108.5
C4—C3—H3	120.1	C9—C8—H8	108.5
C2—C3—H3	120.1	C7—C8—H8	108.5
C3—C4—C5	120.2 (2)	N1—C9—N4	107.73 (14)
C3—C4—H4	119.9	N1—C9—C8	129.97 (14)
C5—C4—H4	119.9	N4—C9—C8	122.28 (13)
C6—C5—C4	120.6 (2)	C9—N1—N2	106.30 (13)
C6—C5—H5	119.7	N3—N2—N1	110.34 (14)
C4—C5—H5	119.7	N2—N3—N4	106.06 (14)
C5—C6—C1	118.65 (18)	C9—N4—N3	109.57 (14)
C5—C6—C7	119.30 (16)	C9—N4—H4A	130.6 (17)
C1—C6—C7	122.05 (16)	N3—N4—H4A	119.8 (18)
N5—C7—C6	112.25 (15)	N6—N5—C7	112.32 (16)
N5—C7—C8	107.41 (14)	N7—N6—N5	174.5 (2)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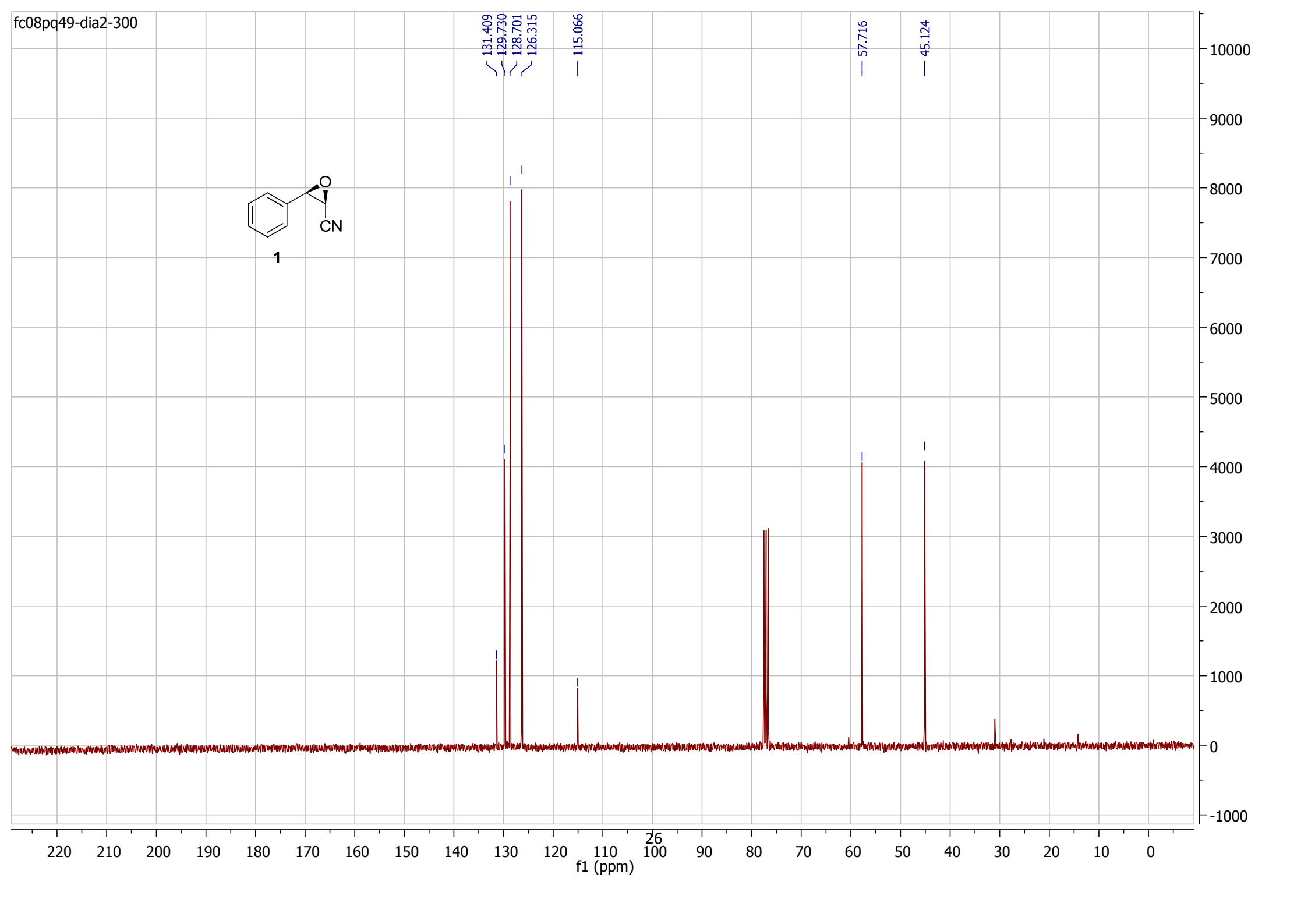
O
CN
1
5.06
1.02
1.00
f1 (ppm)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CN
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8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
220
210
200
190
180
170
160
150
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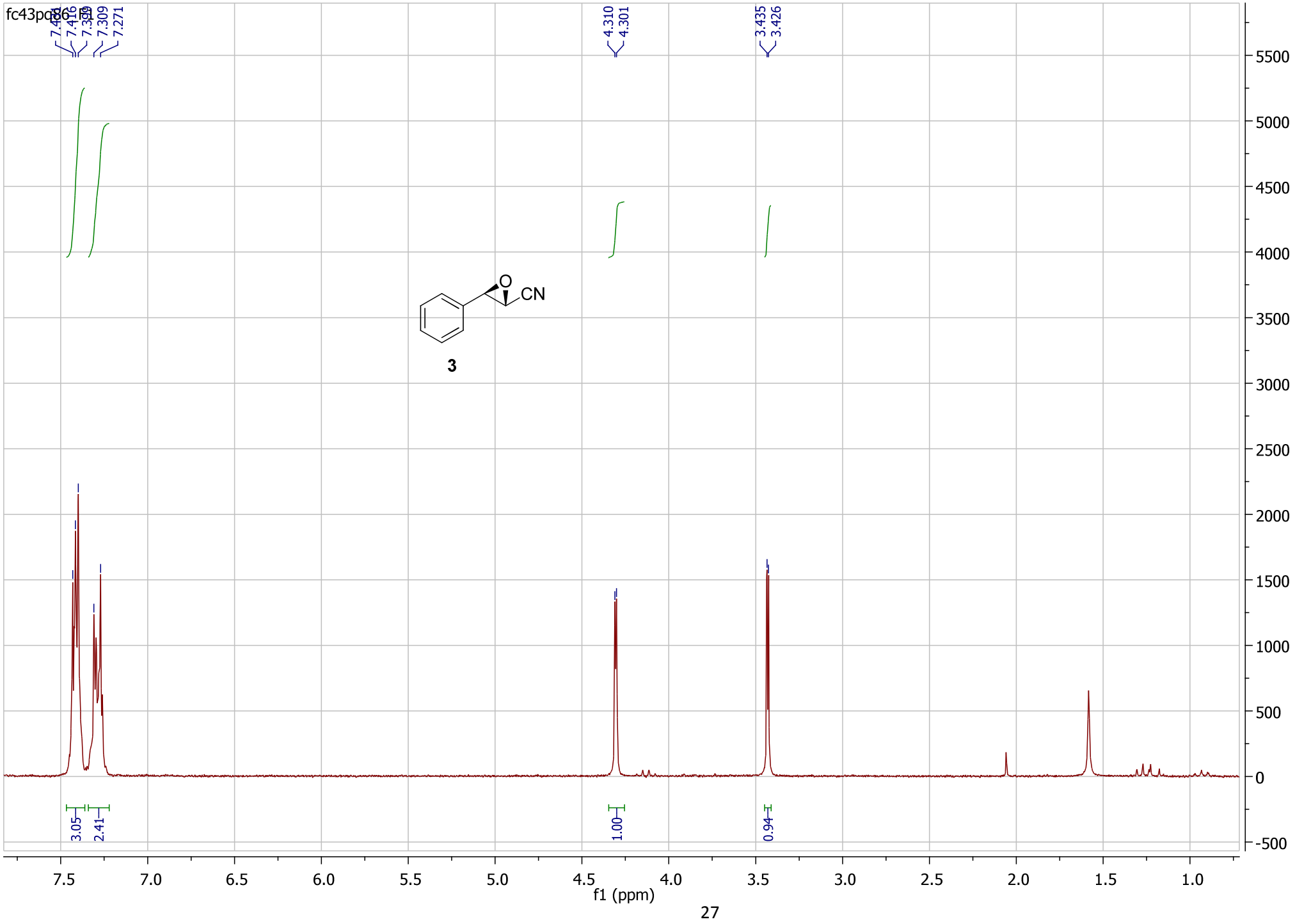
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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3.426
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CN
3
3.05
2.41
1.00
0.94
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
7.5
7.0
6.5
6.0
5.5
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4.5
4.0
3.5
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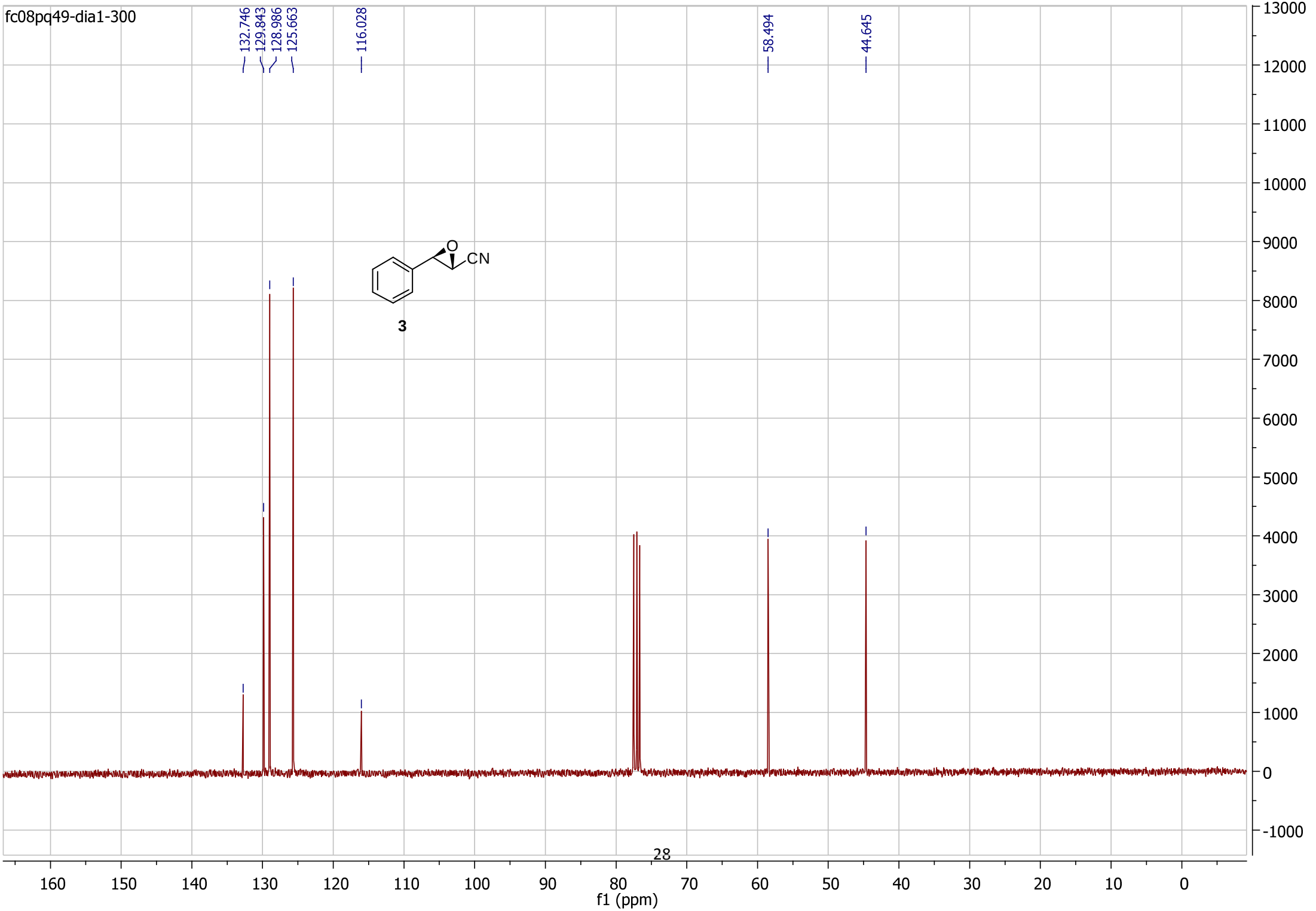
2.0
1.5
1.0
f1 (ppm)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CN
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28
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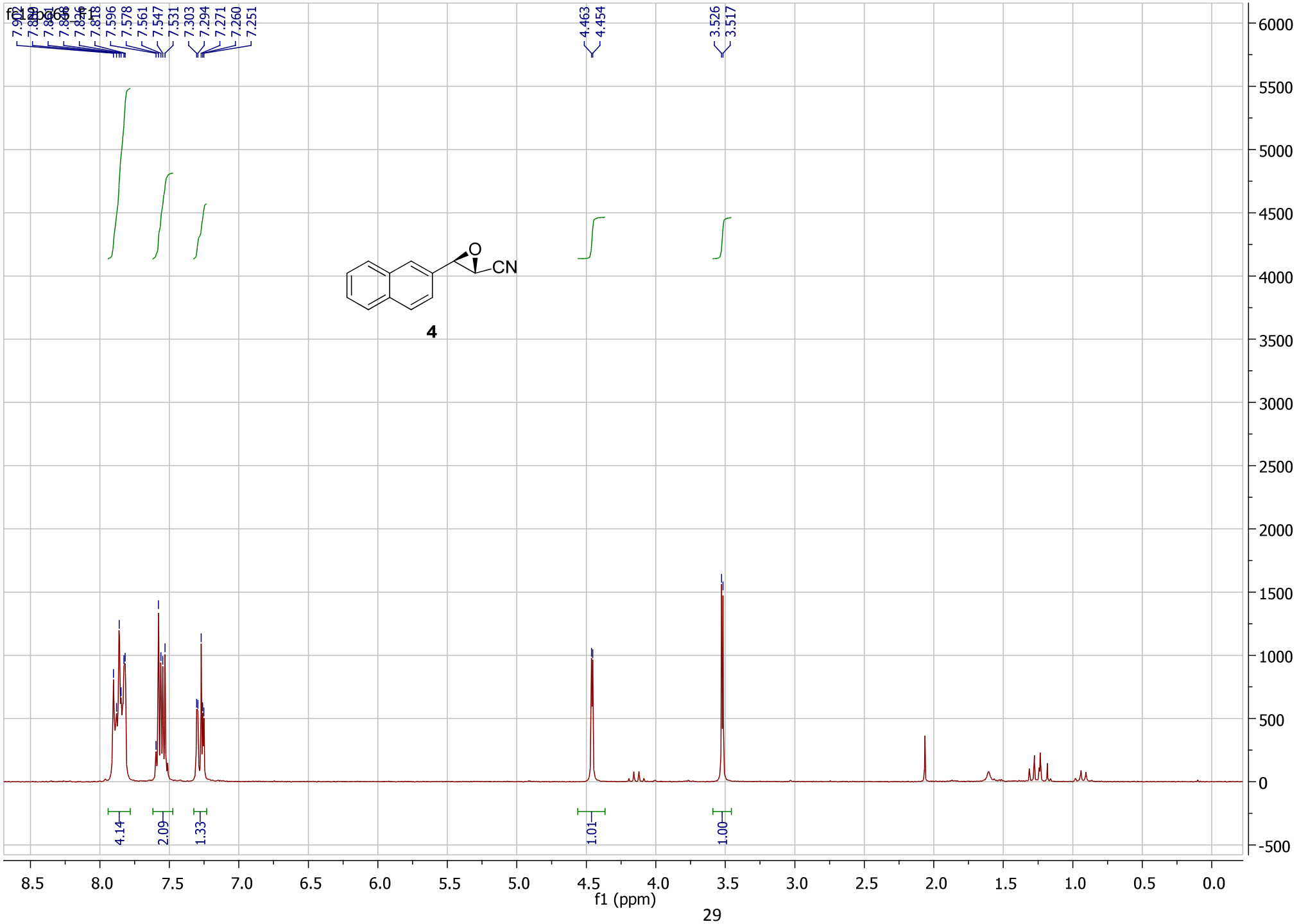
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7.561
7.547
7.531
7.303
7.294
7.271
7.260
7.251
4.463
4.454
3.526
3.517
O
CN
4
4.14
2.09
1.33
1.01
1.00
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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1.0
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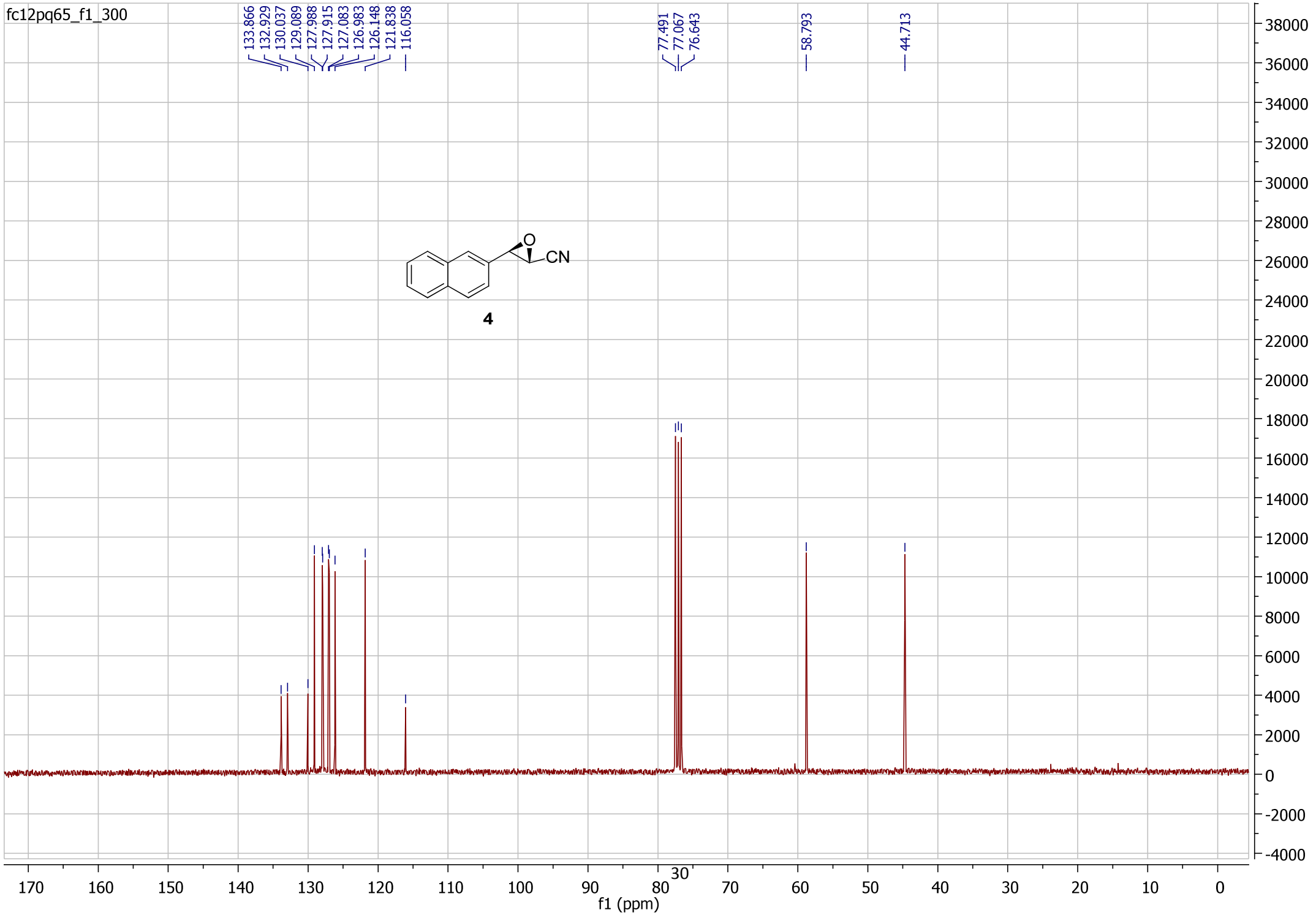
f1 (ppm)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76.643
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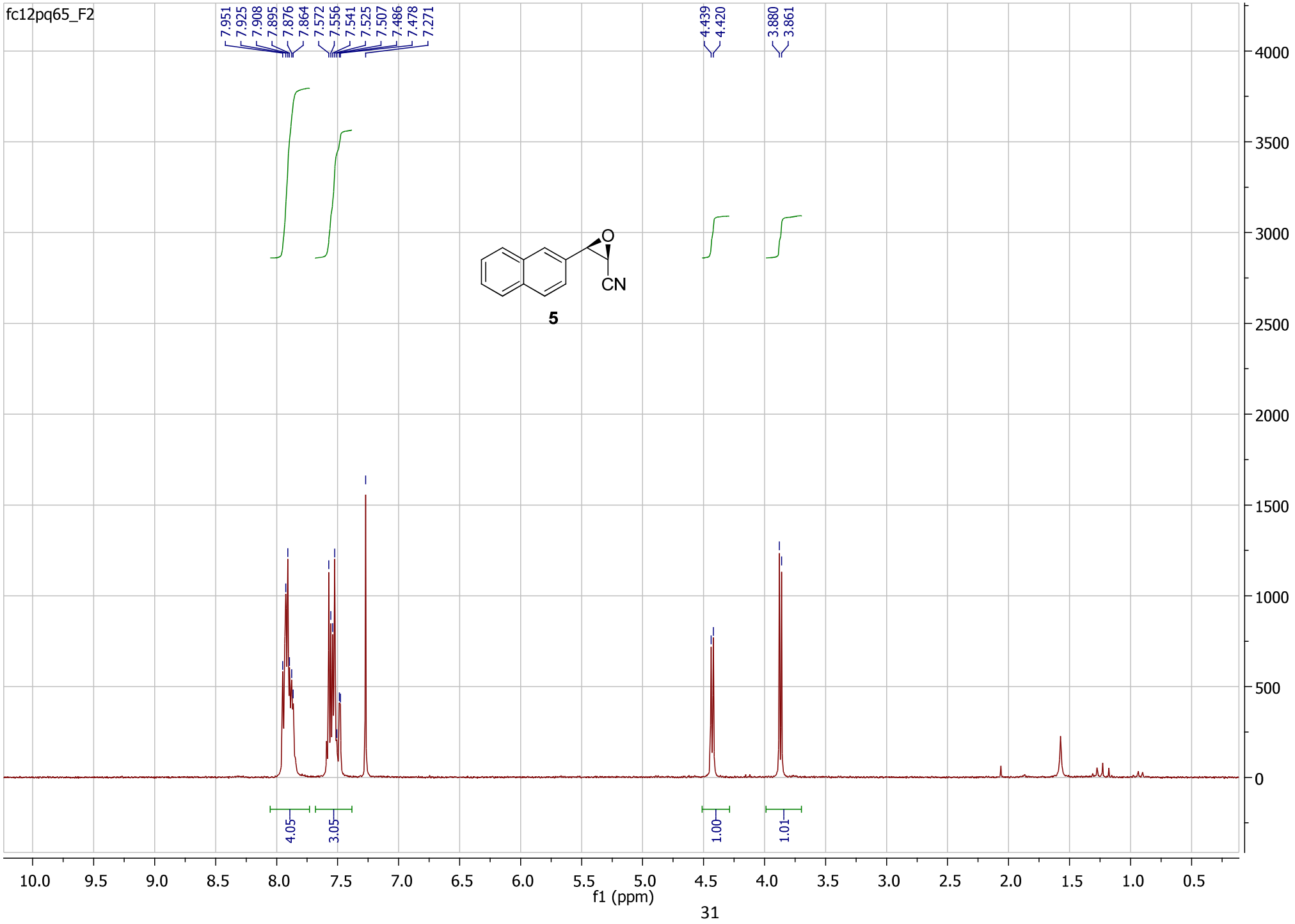
O
CN
4
f1 (ppm)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7.864
7.572
7.556
7.541
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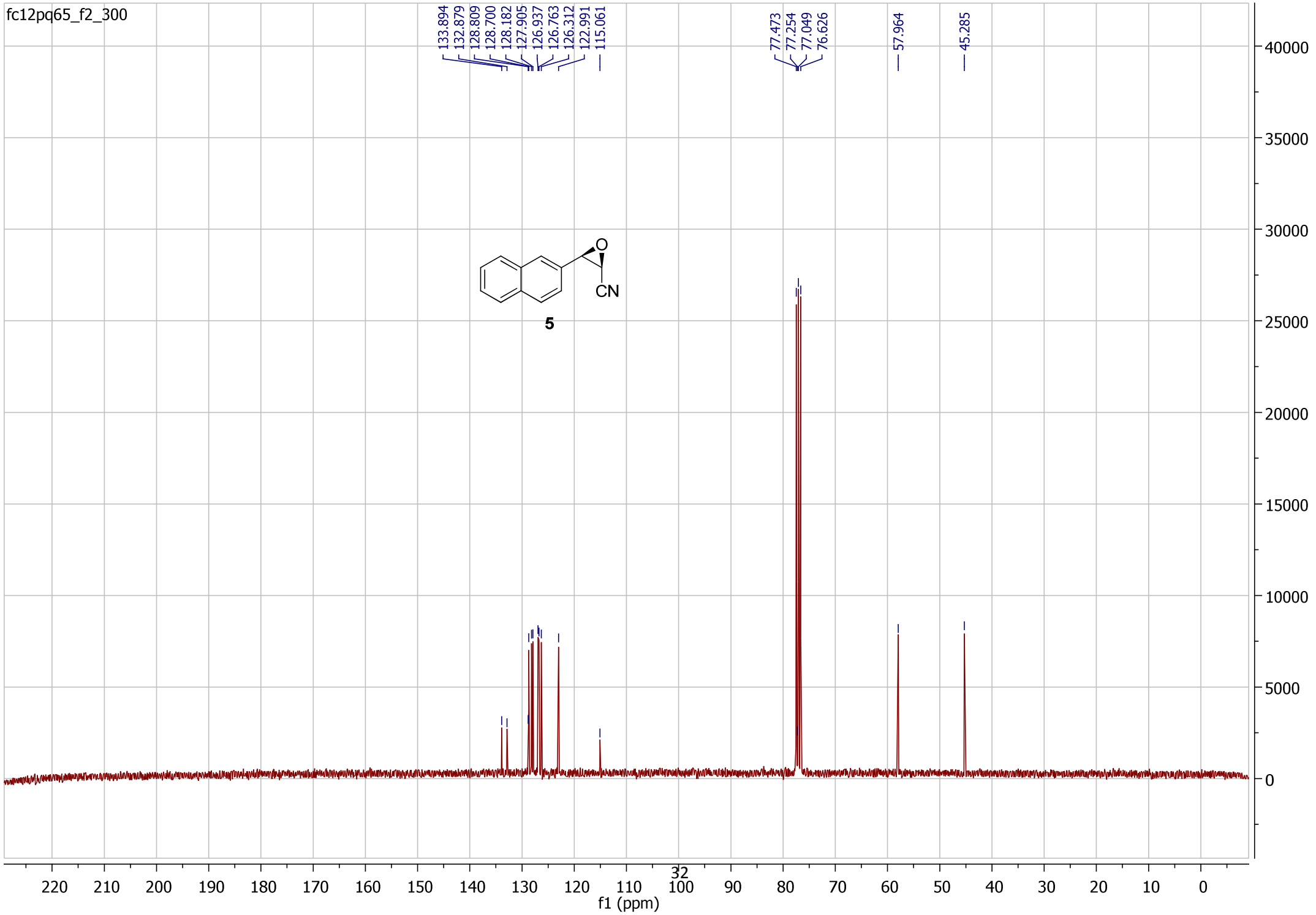
7.486
7.478
7.271
4.439
4.420
3.880
3.861
O
CN
5
4.05
3.05
1.00
1.01
4000
3500
3000
2500
2000
1500
1000
500
0
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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77.254
77.049
76.626
57.964
45.285
O
CN
5
40000
35000
30000
25000
20000
15000
10000
5000
0
220
210
200
190
180
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120
110
100
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20
10
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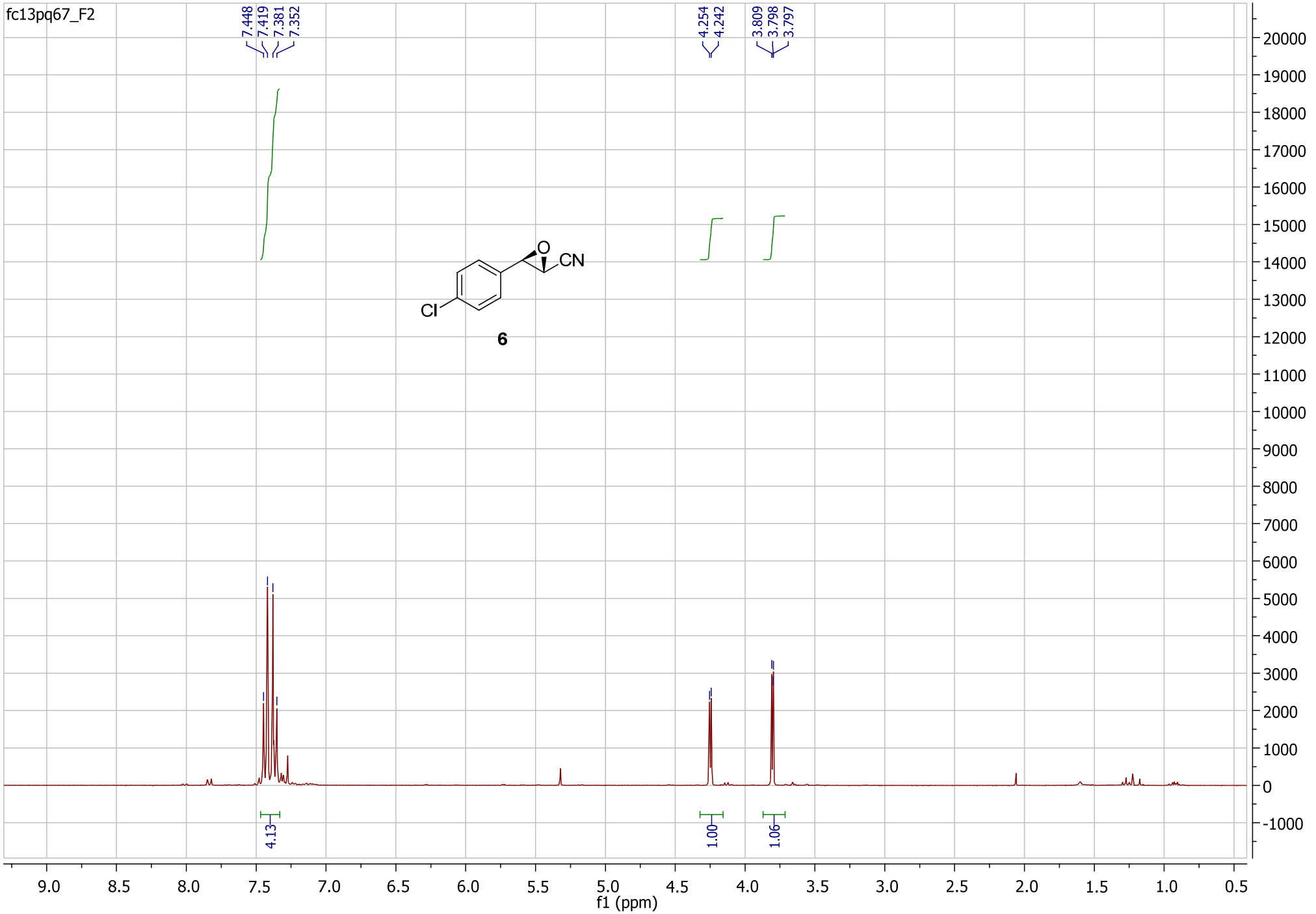
f1 (ppm)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7.352
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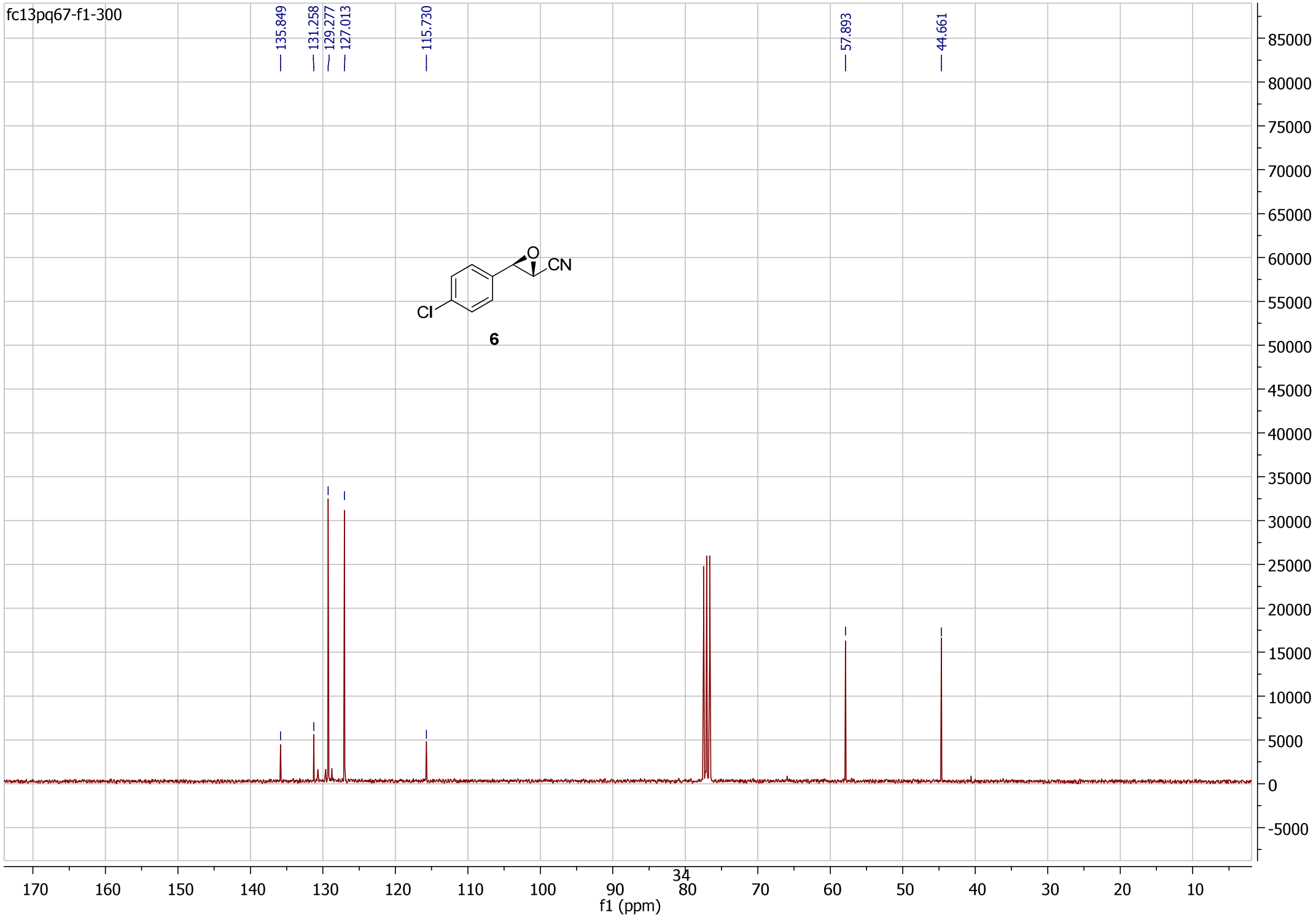
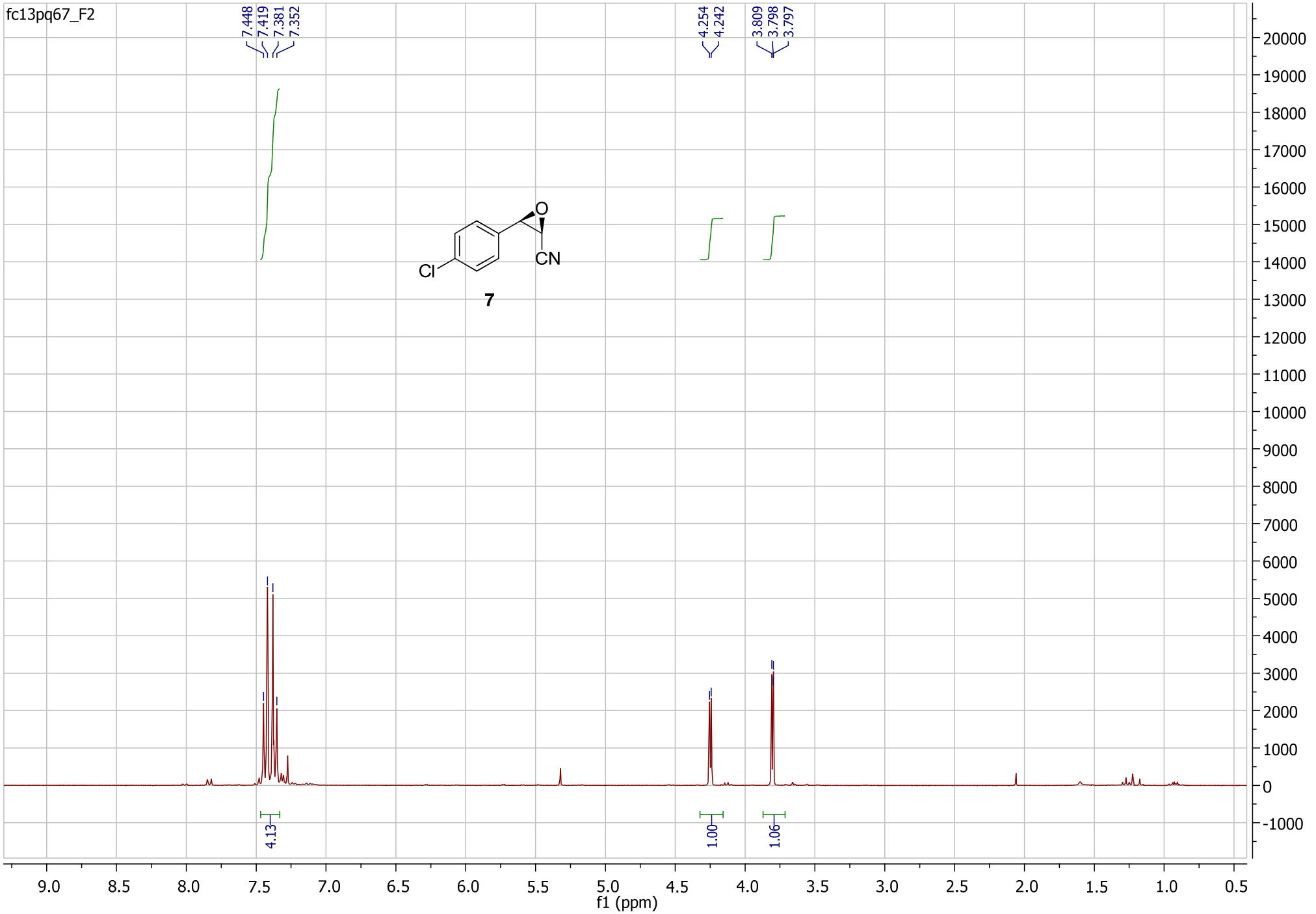
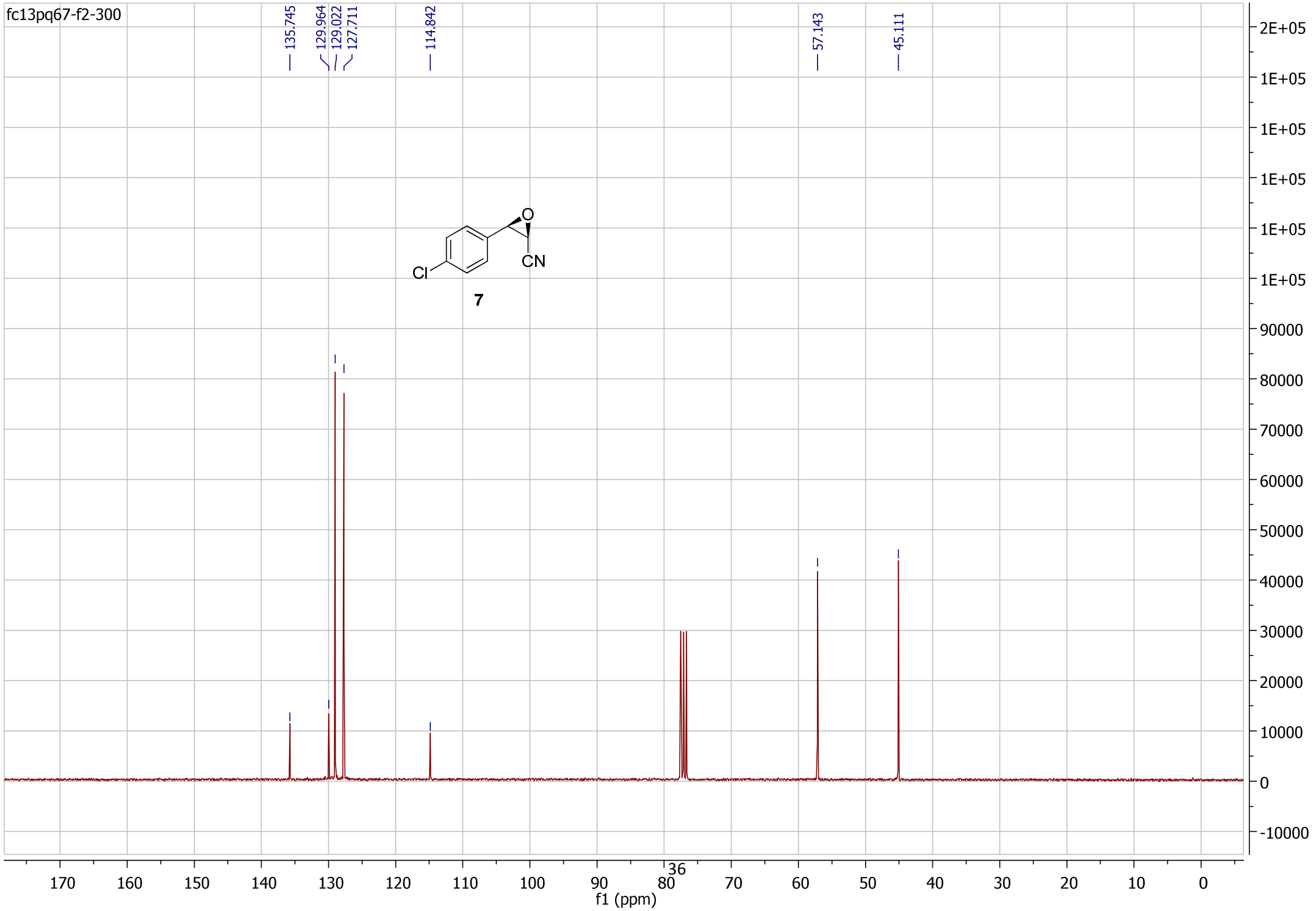
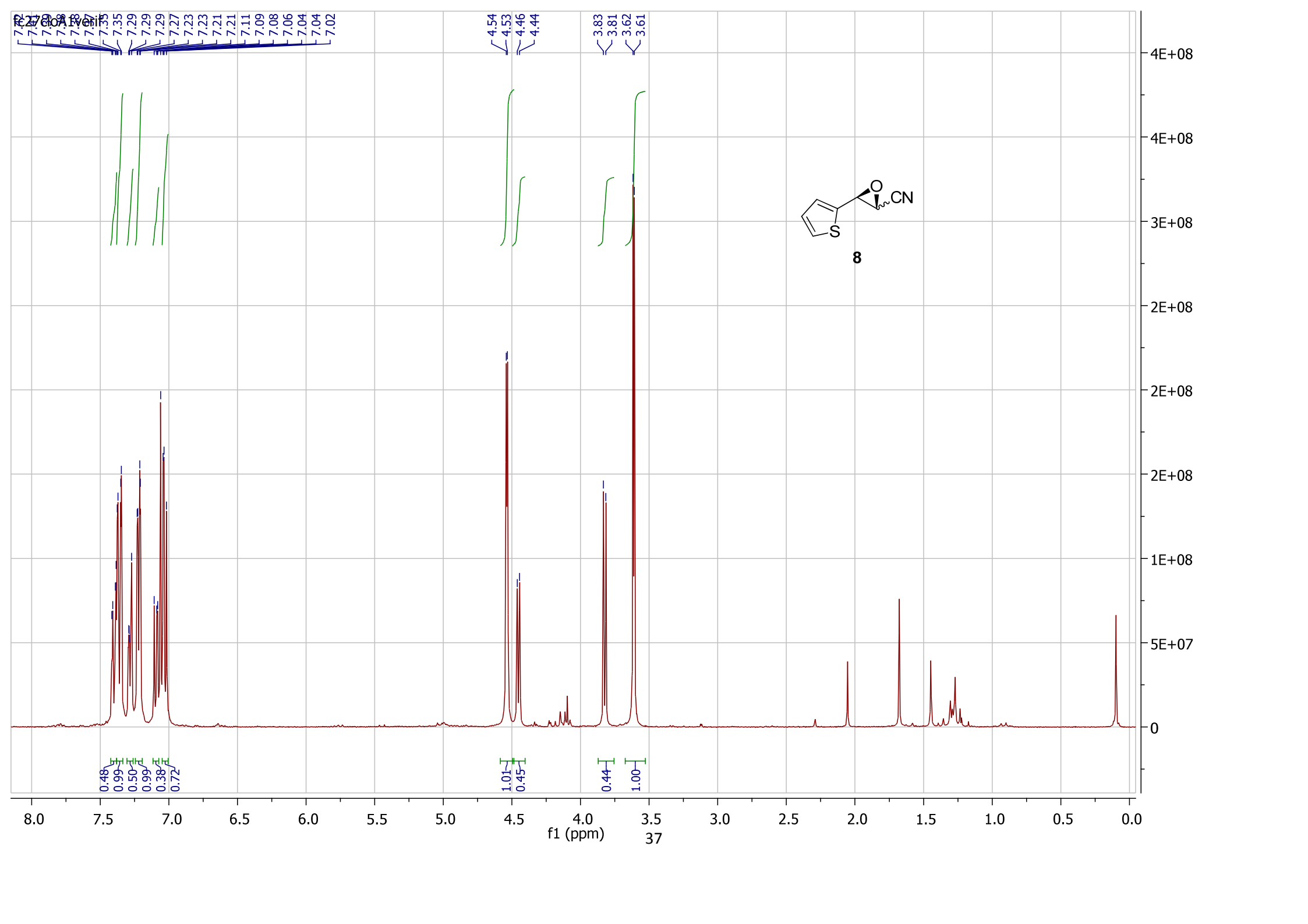
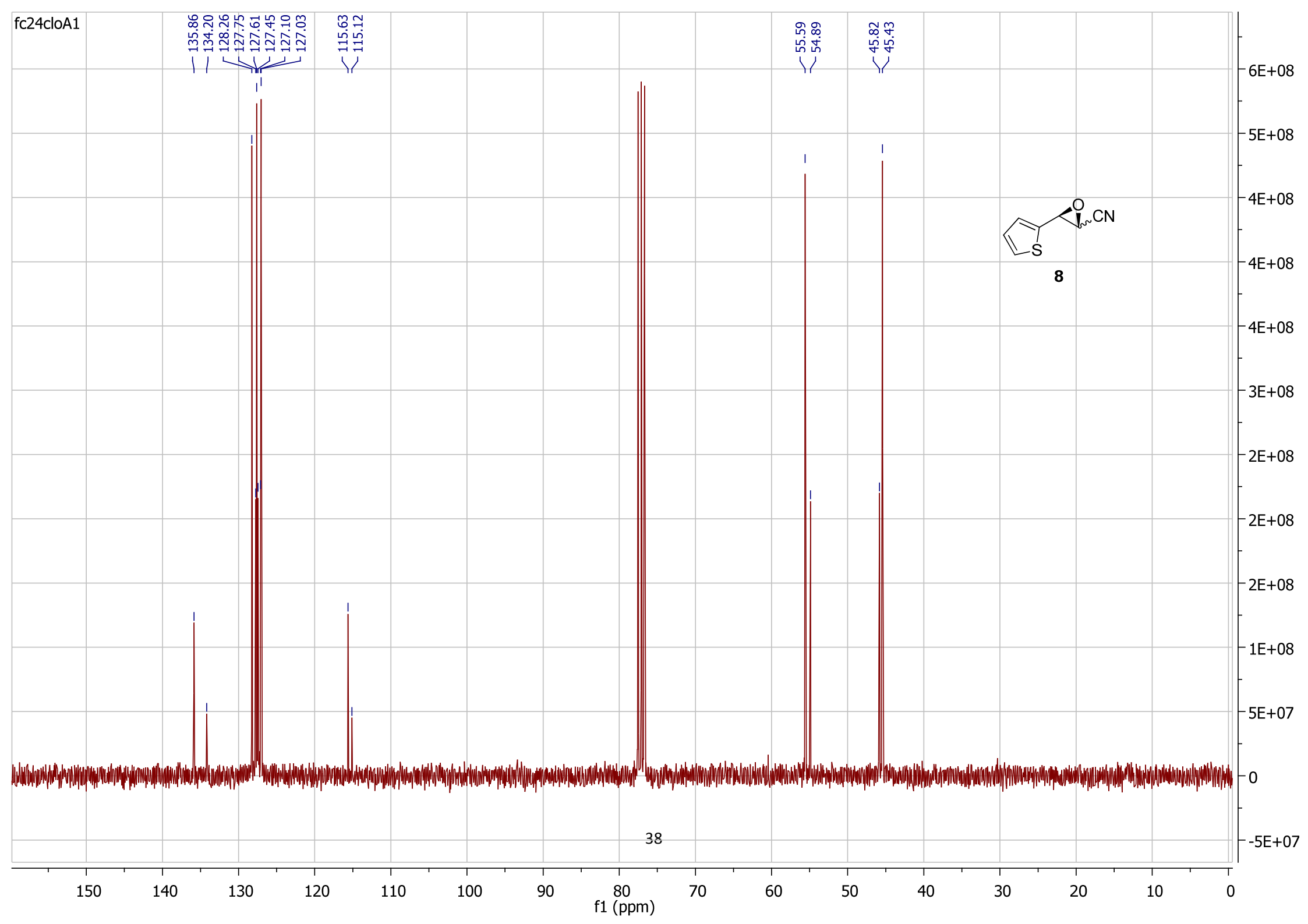
4.242
3.809
3.798
3.797
O
CN
Cl
6
4.13
1.00
1.06
f1 (ppm)

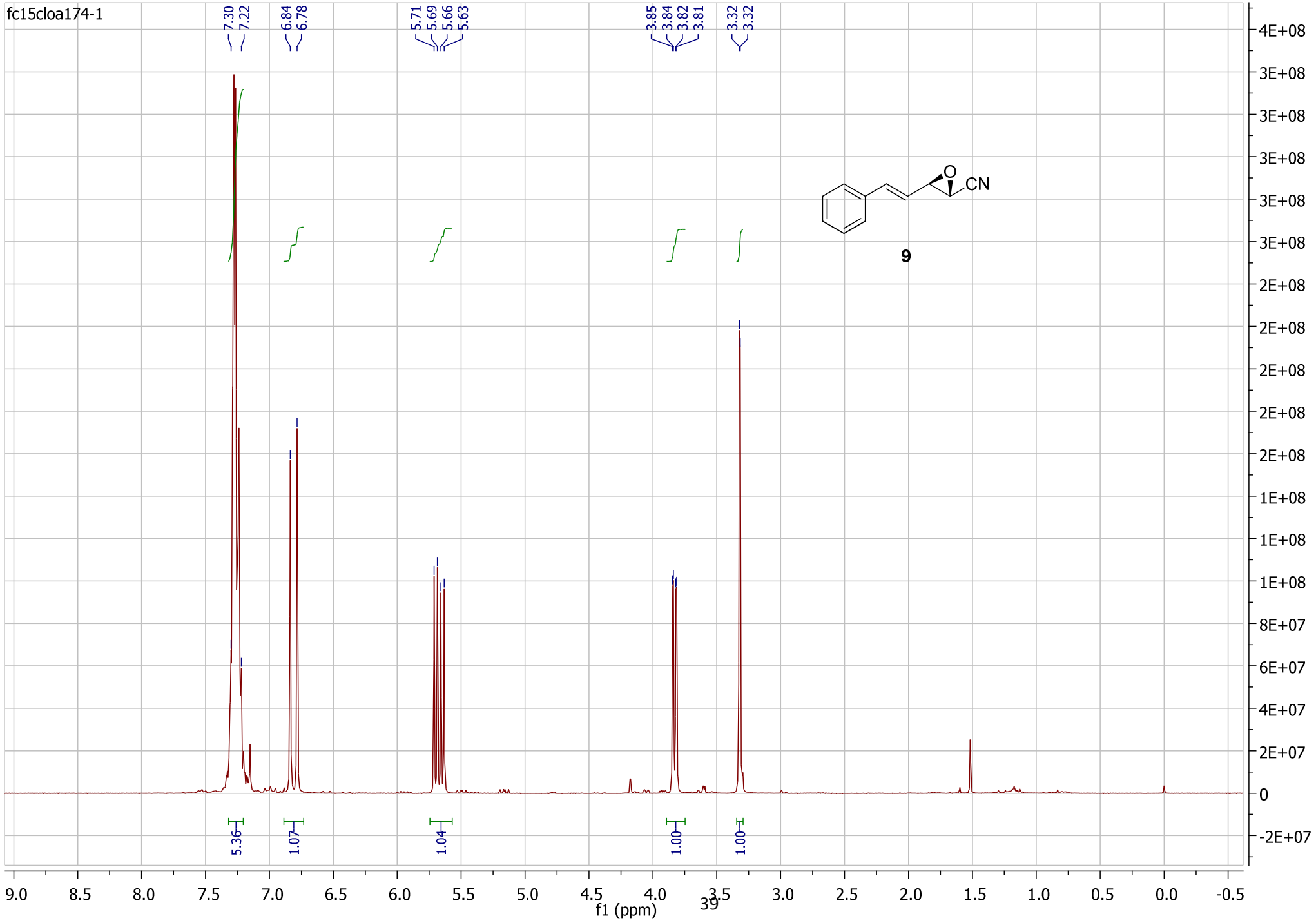

fc13pq67-f1-300
135.849
131.258
129.277
127.013
115.730
57.893
44.661
O
CN
Cl
6
85000
80000
75000
70000
65000
60000
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

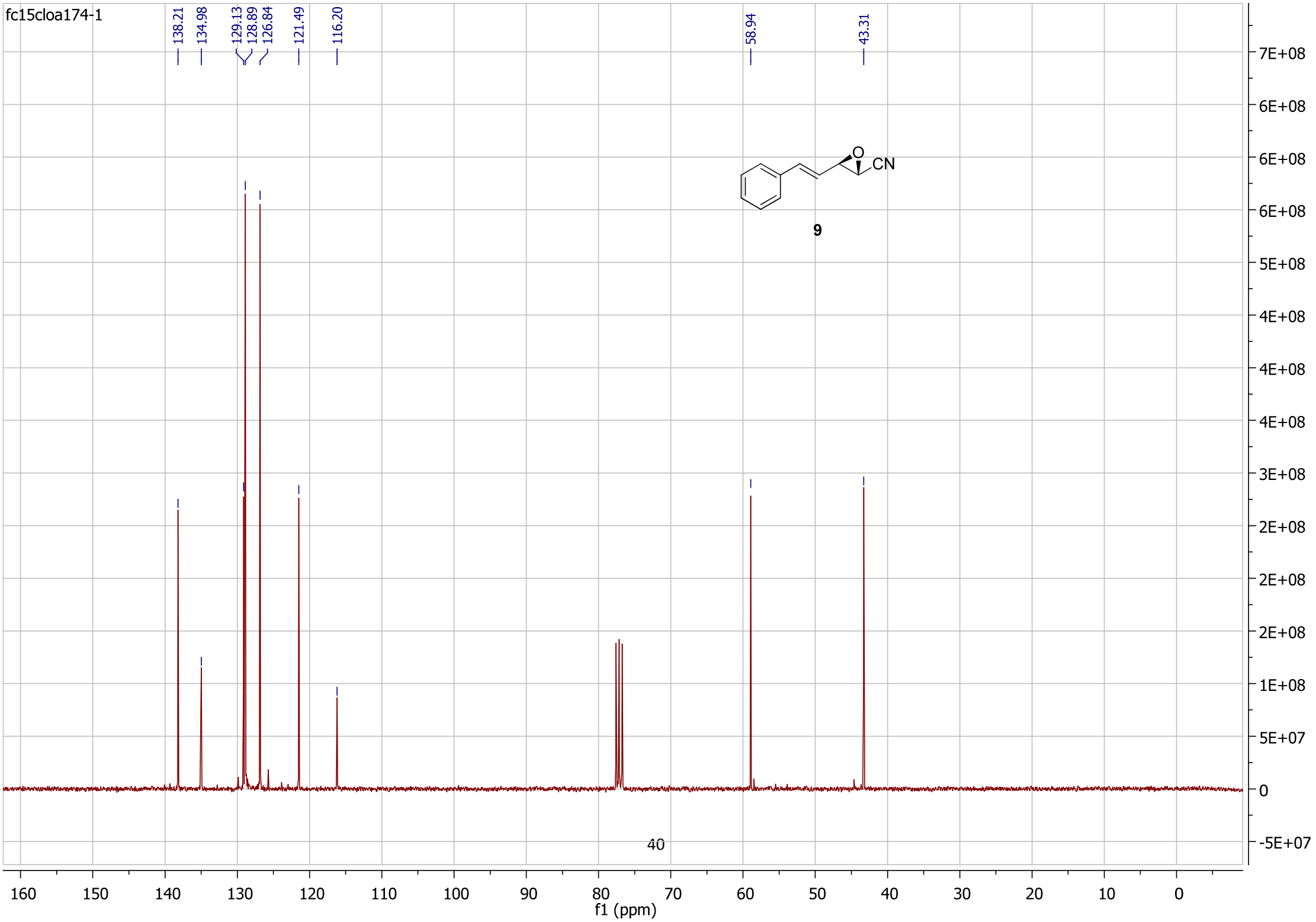

fc13pq67_F2
7.448
7.419
7.381
7.352
4.254
4.242
3.809
3.798
3.797
O
Cl
CN
7
4.13
1.00
1.06
f1 (ppm)

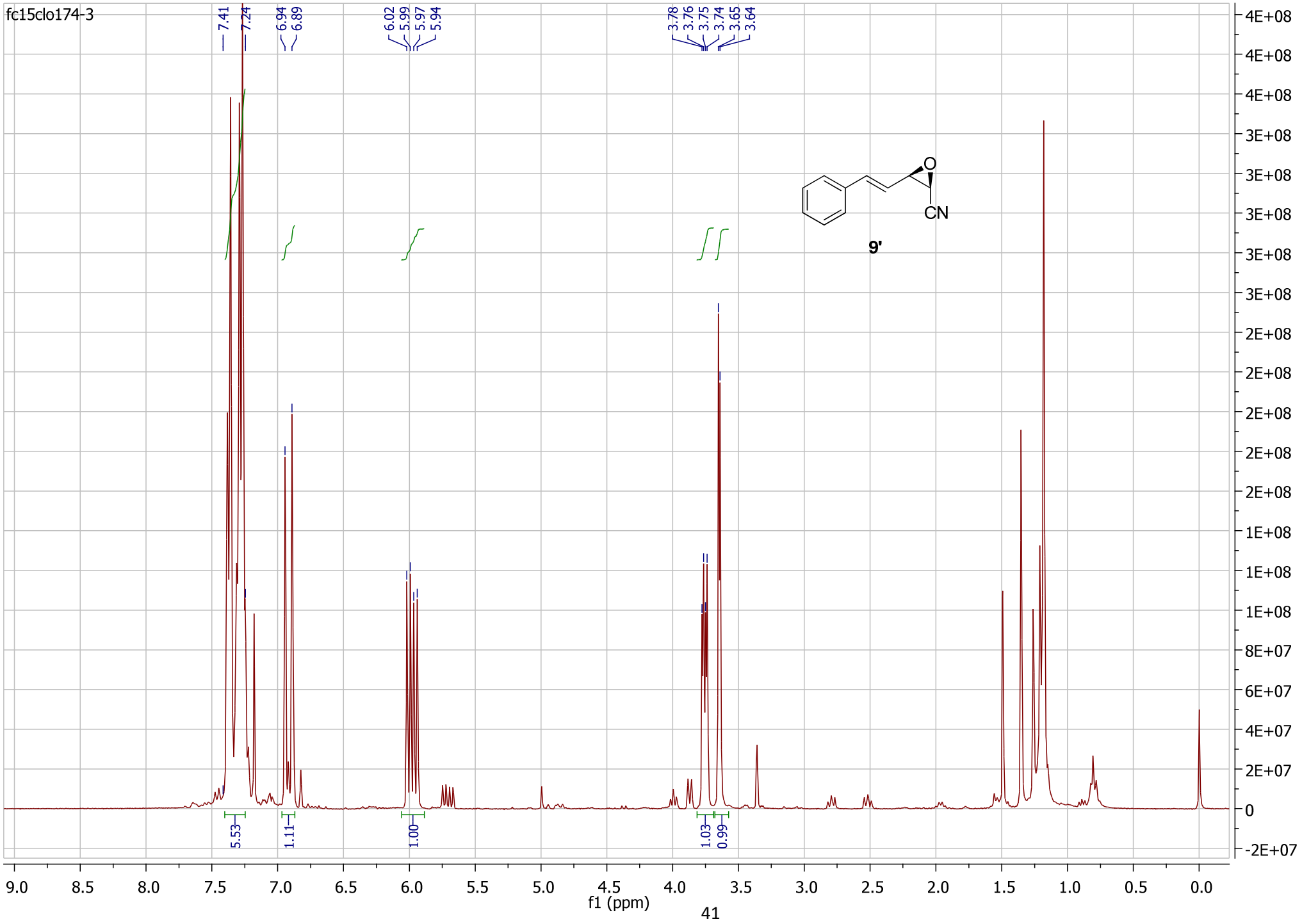

fc13pq67-f2-300
135.745
129.964
129.022
127.711
114.842
57.143
45.111
O
CN
Cl
7
2E+05
1E+05
1E+05
1E+05
1E+05
1E+05
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

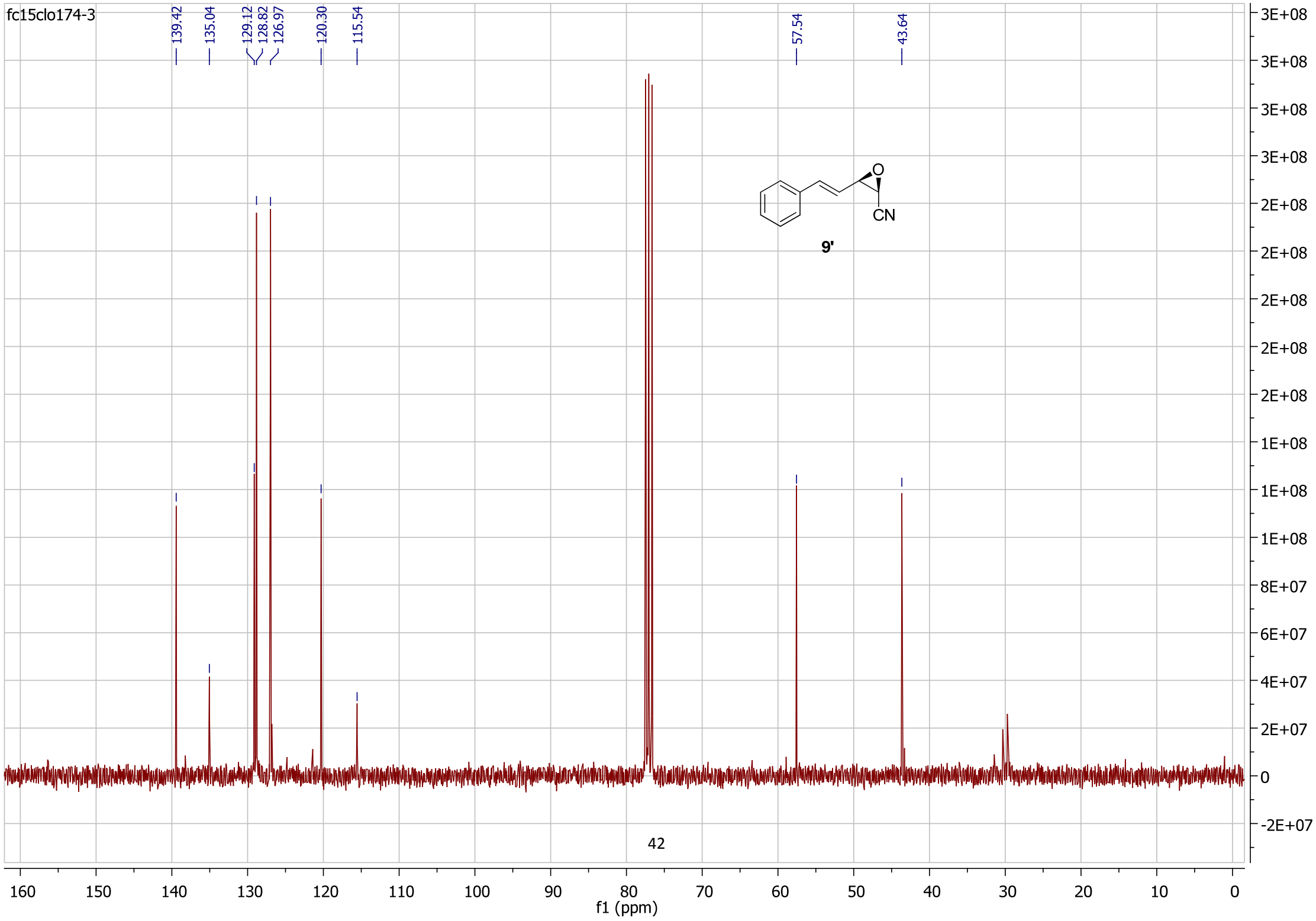

fc276oA1ver1
7.35
7.29
7.29
7.29
7.27
7.23
7.23
7.21
7.21
7.11
7.09
7.08
7.06
7.04
7.04
7.02
4.54
4.53
4.46
4.44
3.83
3.81
3.62
3.61
O
CN
S
8
0.48
0.99
0.50
0.99
0.38
0.72
1.01
0.45
0.44
1.00
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

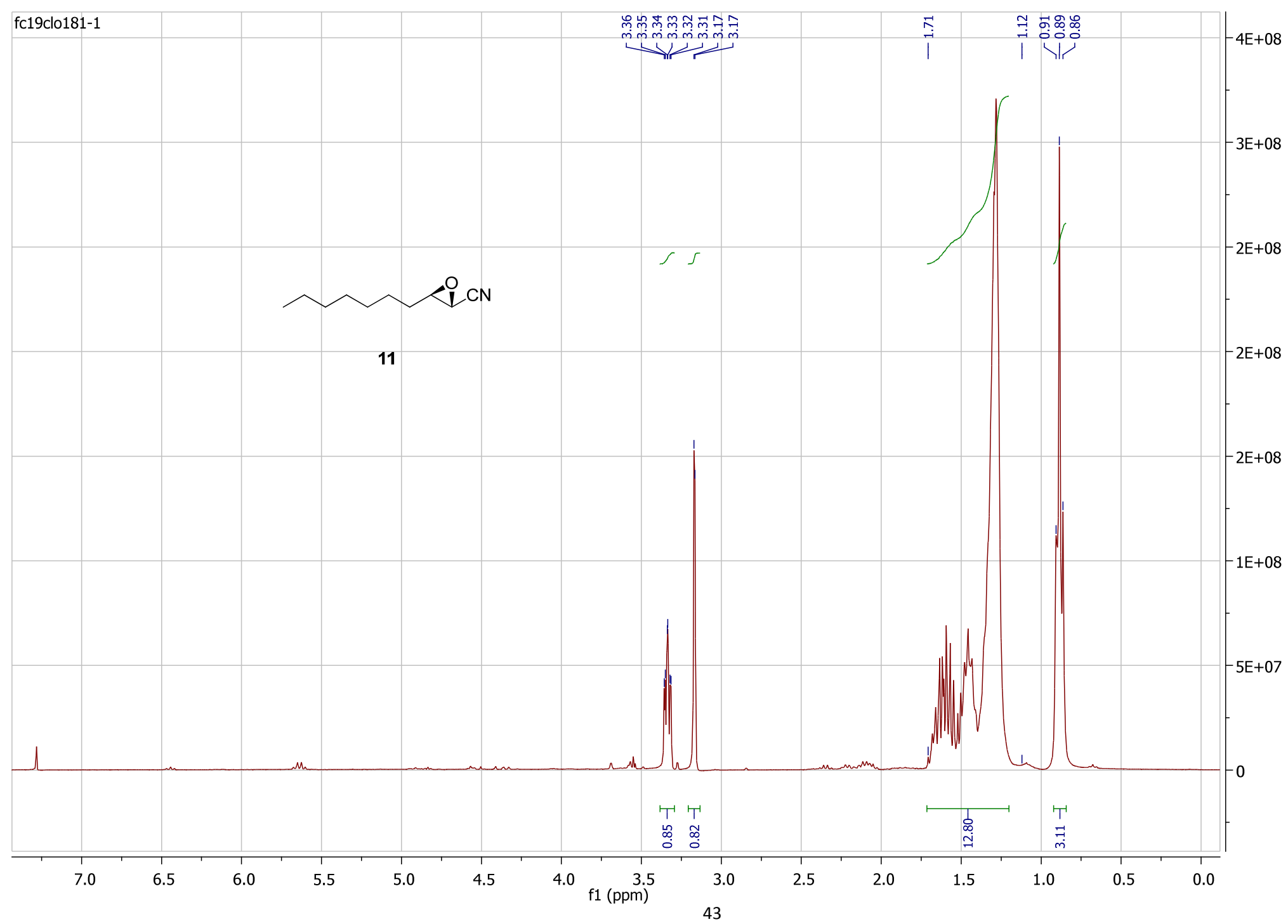

fc24cloA1
135.86
134.20
128.26
127.75
127.61
127.45
127.10
127.03
115.63
115.12
55.59
54.89
45.82
45.43
O
CN
S
8
38
6E+08
5E+08
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
-5E+07
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

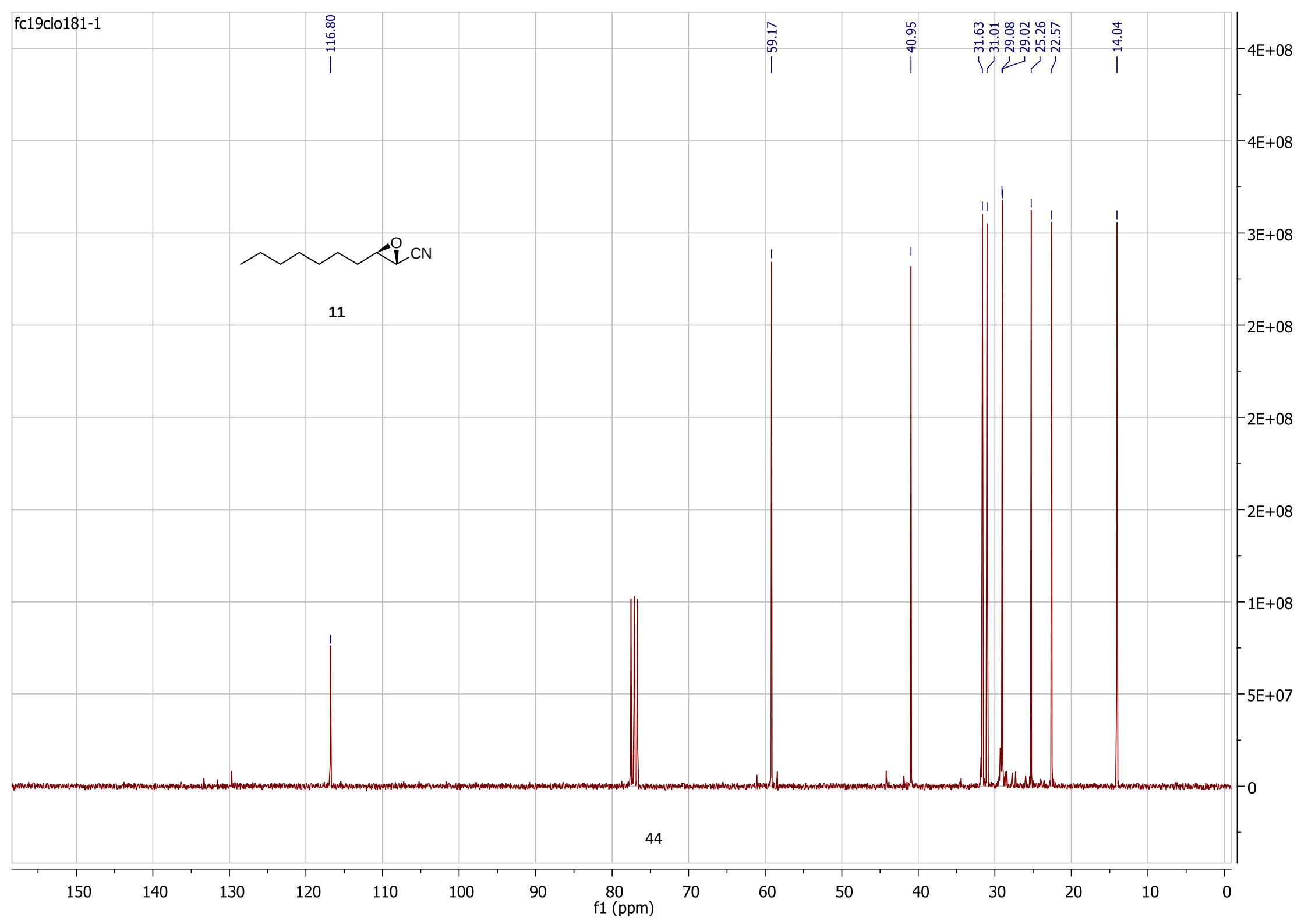

fc15cloa174-1
7.30
7.22
6.84
6.78
5.71
5.69
5.66
5.63
3.85
3.84
3.82
3.81
3.32
3.32
O
CN
9
5.36
1.07
1.04
1.00
1.00
4E+08
3E+08
3E+08
3E+08
3E+08
3E+08
2E+08
2E+08
2E+08
2E+08
2E+08
1E+08
1E+08
1E+08
8E+07
6E+07
4E+07
2E+07
0
-2E+07
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

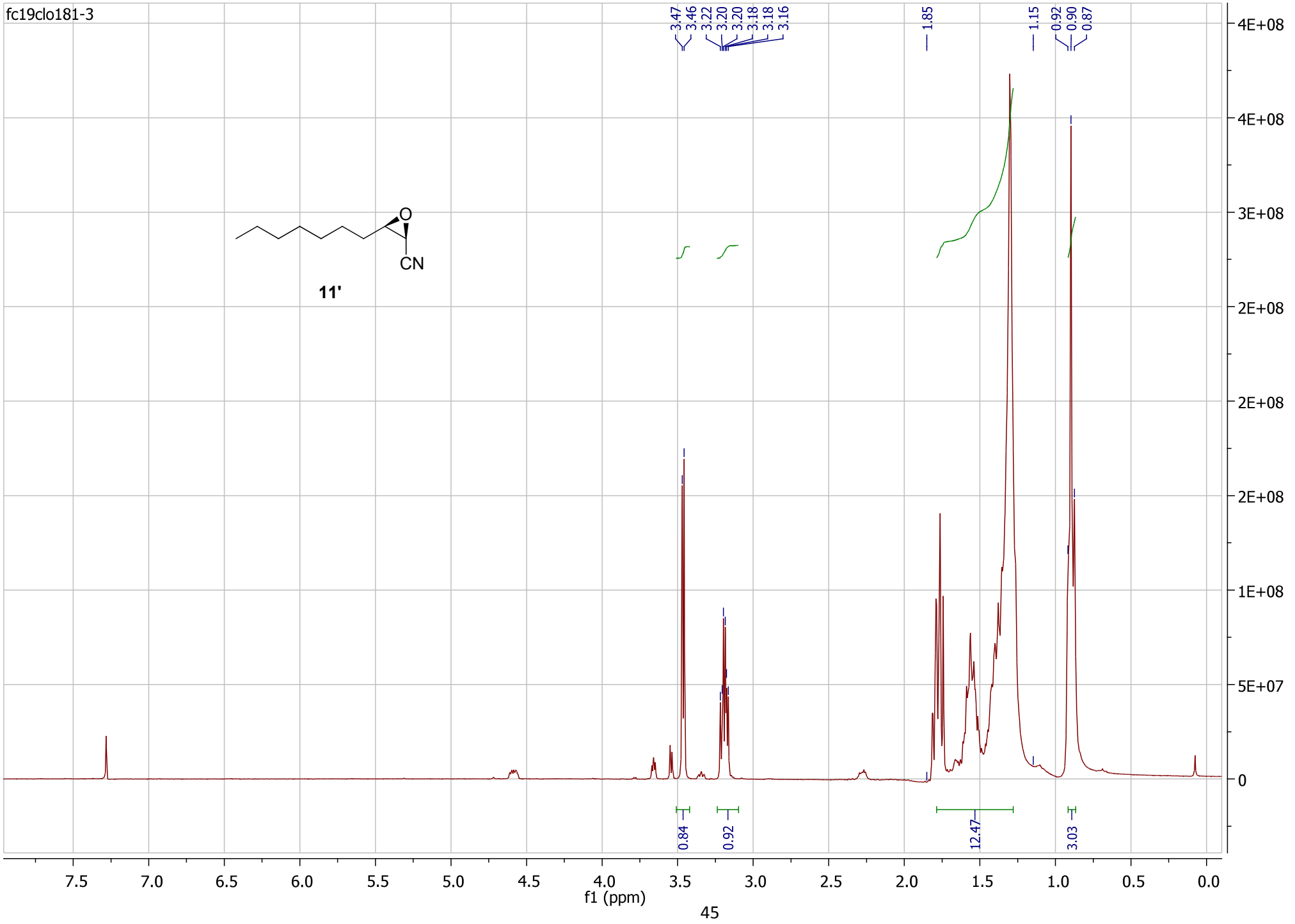

fc15cloa174-1
138.21
134.98
129.13
128.89
126.84
121.49
116.20
58.94
43.31
O
CN
9
40
7E+08
6E+08
6E+08
6E+08
5E+08
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
-5E+07
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

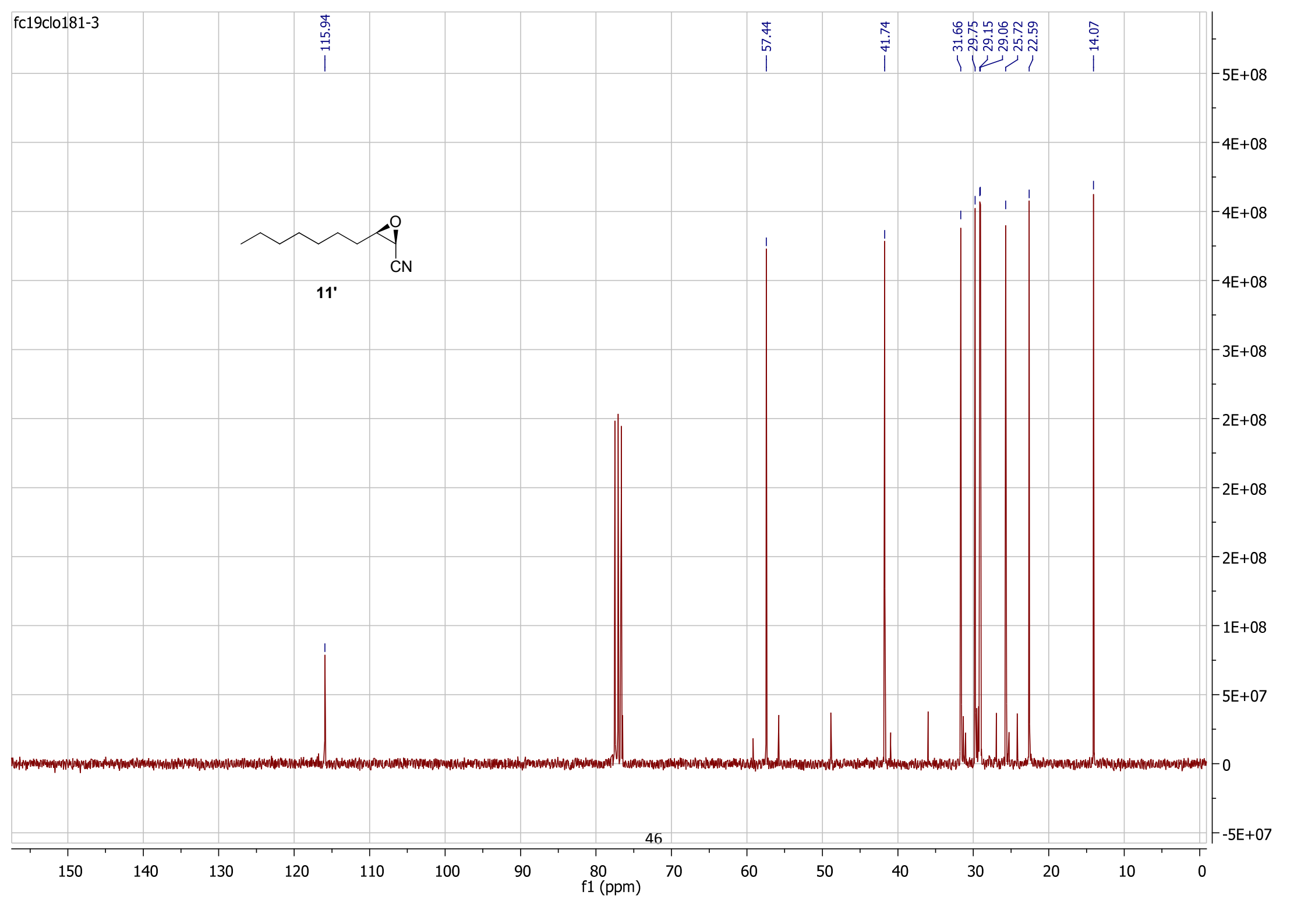

fc15clo174-3
7.41
7.24
6.94
6.89
6.02
5.99
5.97
5.94
3.78
3.76
3.75
3.74
3.65
3.64
O
CN
9'
5.53
1.11
1.00
1.03
0.99
4E+08
3E+08
2E+08
1E+08
8E+07
6E+07
4E+07
2E+07
0
-2E+07
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

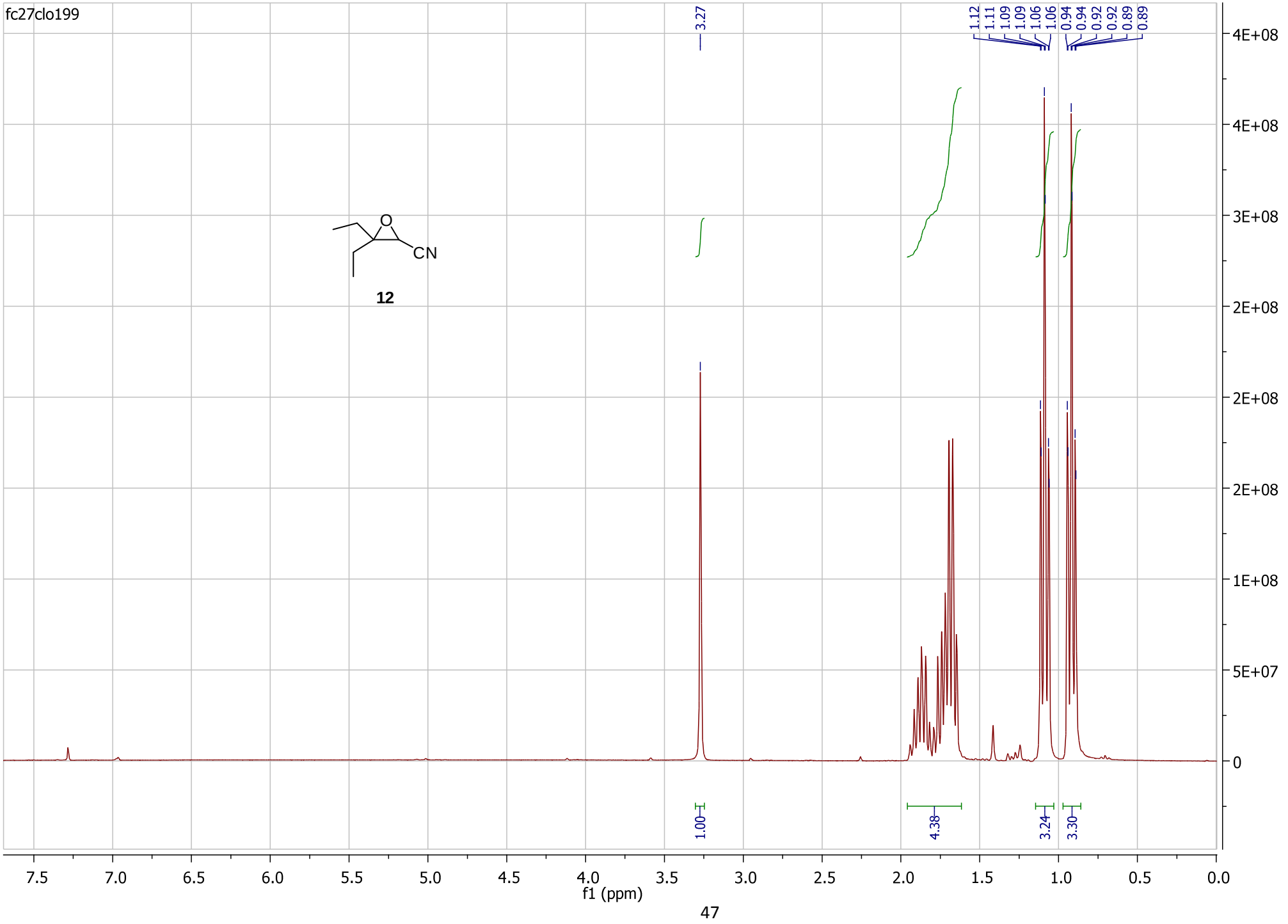

fc15clo174-3
139.42
135.04
129.12
128.82
126.97
120.30
115.54
57.54
43.64
O
CN
9'
42
f1 (ppm)

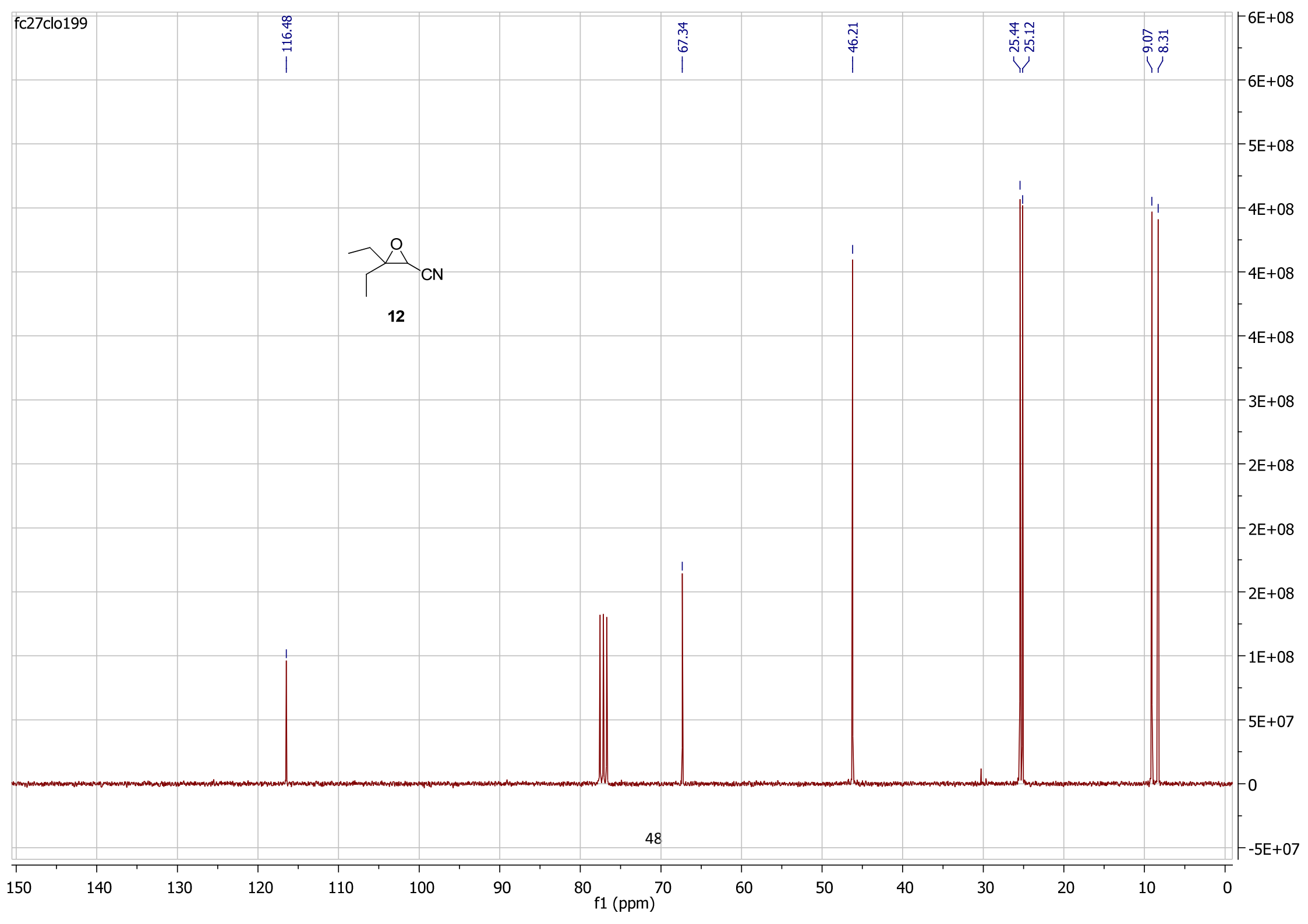

fc19clo181-1
3.36
3.35
3.34
3.33
3.32
3.31
3.17
3.17
1.71
1.12
0.91
0.89
0.86
O
CN
11
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
0.85
0.82
12.80
3.11
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

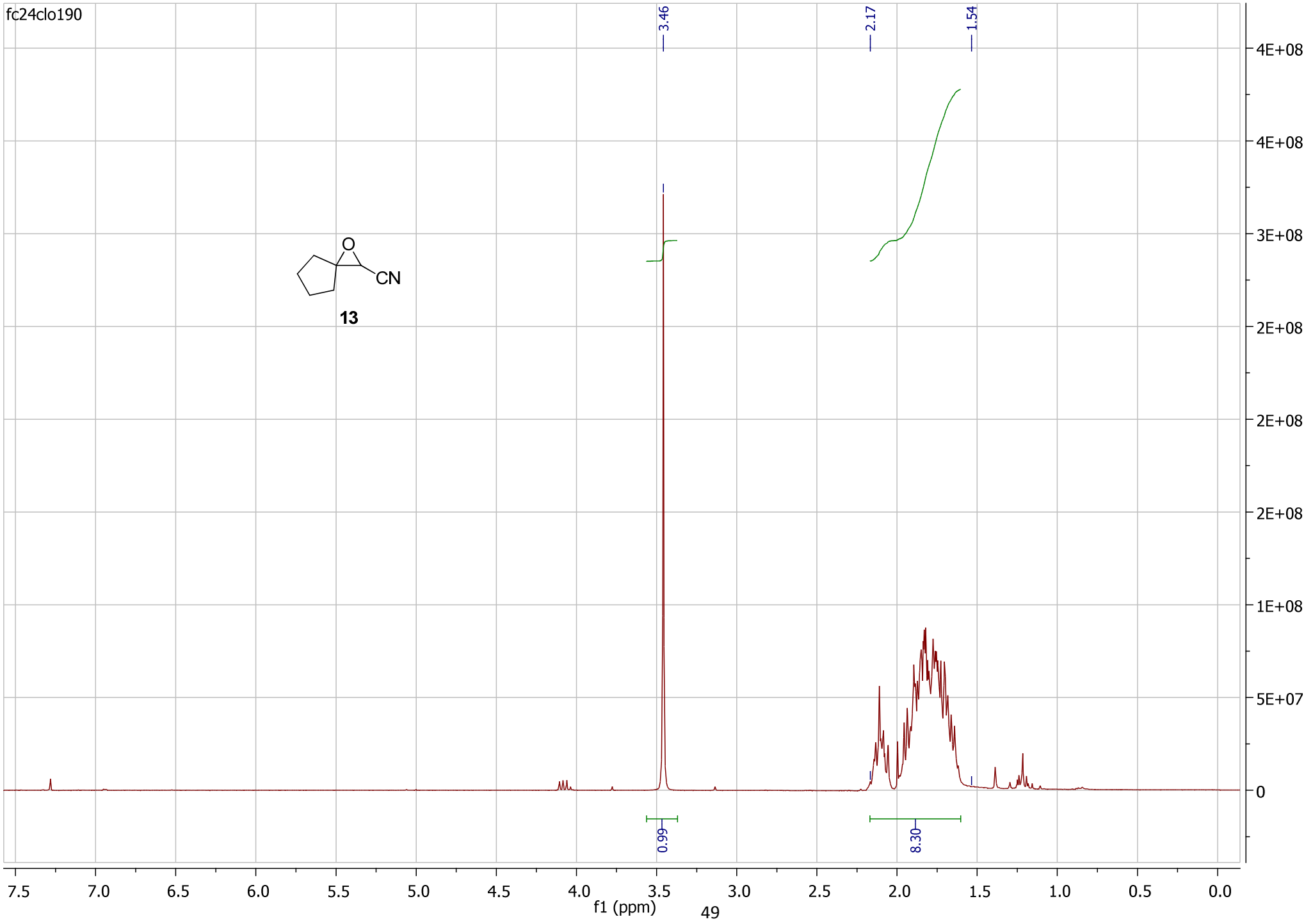

fc19clo181-1
116.80
59.17
40.95
31.63
31.01
29.08
29.02
25.26
22.57
14.04
O
CN
11
44
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

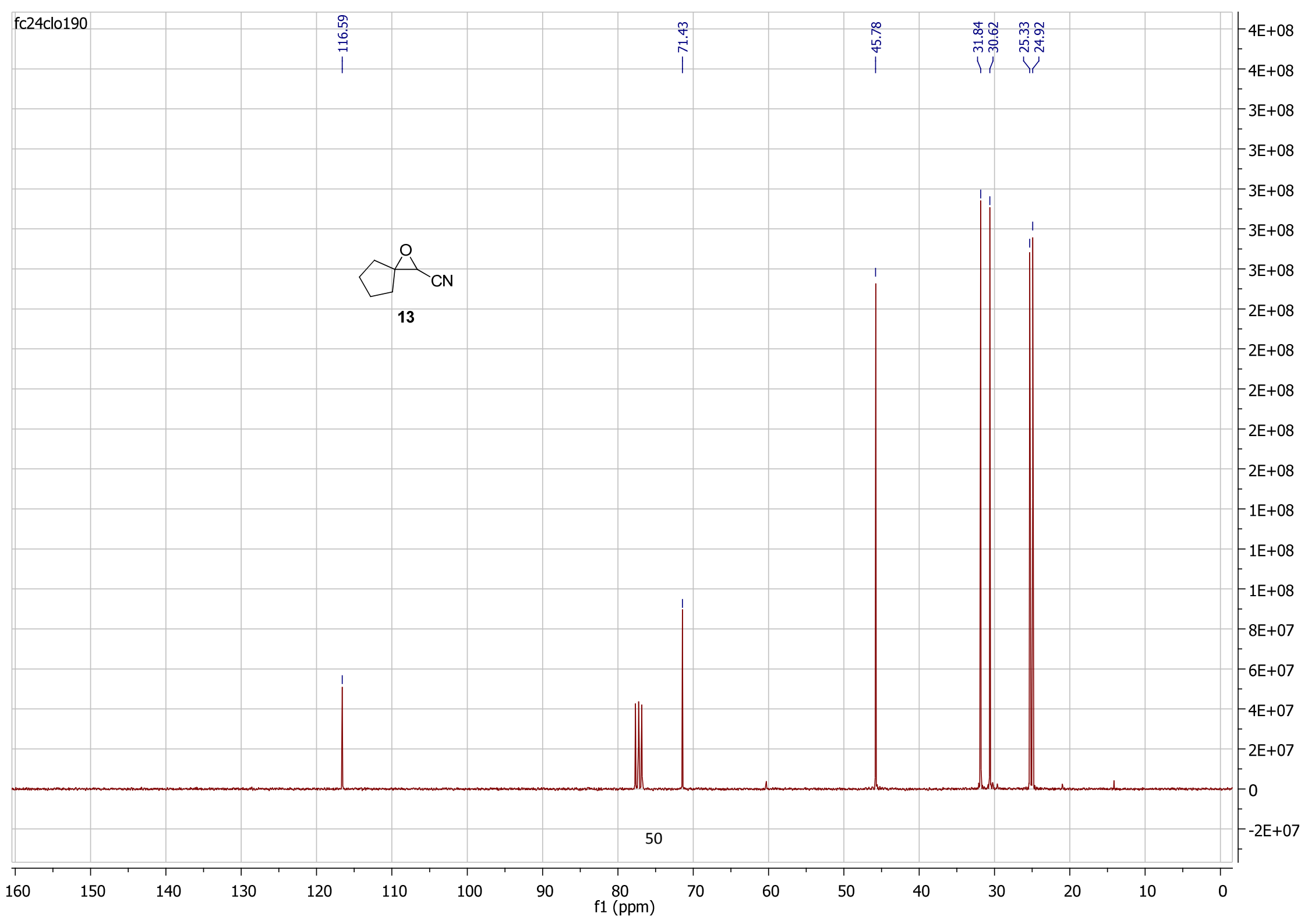

fc19clo181-3
3.47
3.46
3.22
3.20
3.20
3.18
3.18
3.16
1.85
1.15
0.92
0.90
0.87
O
CN
11'
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
0.84
0.92
12.47
3.03
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

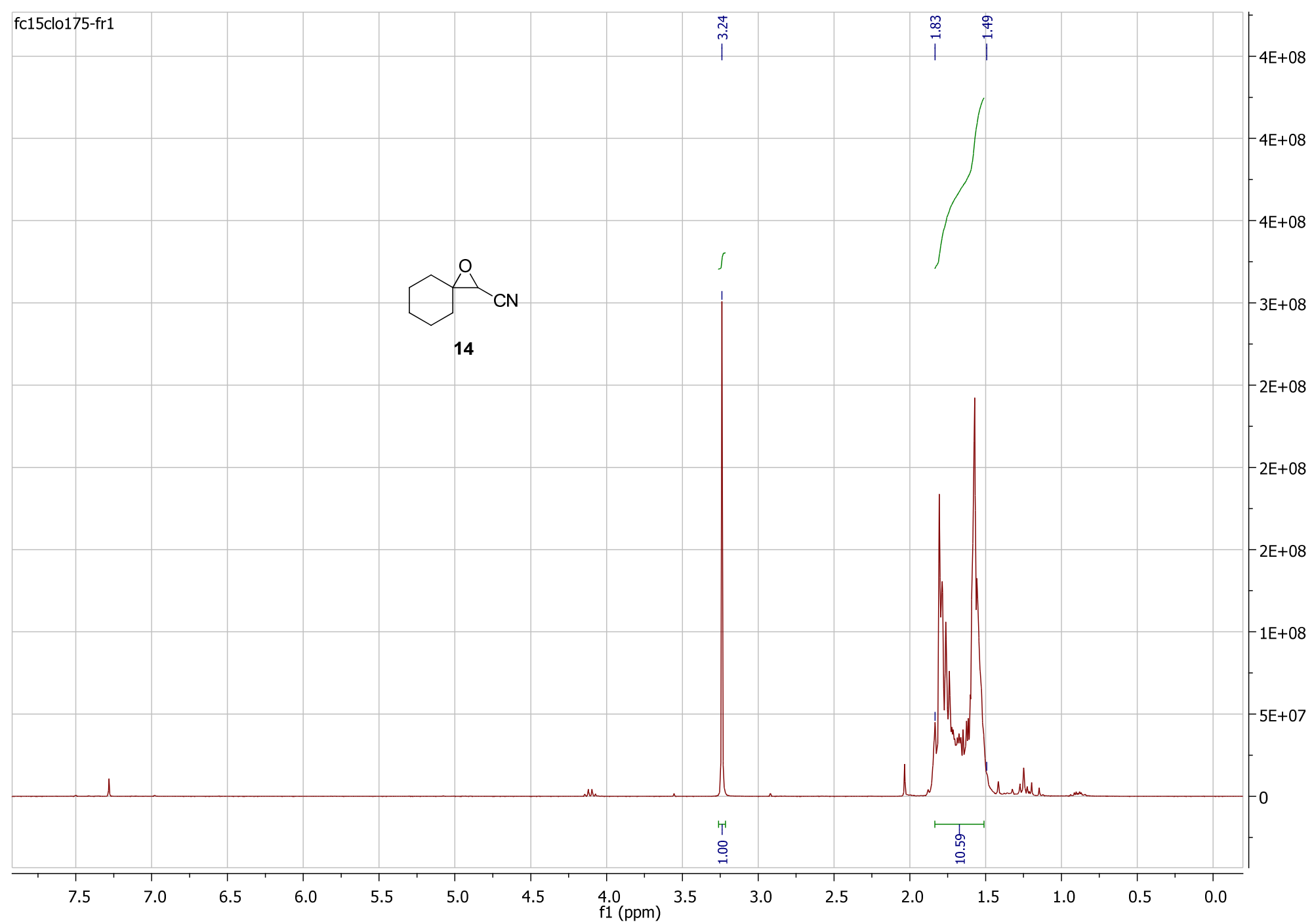

fc19clo181-3
115.94
57.44
41.74
31.66
29.75
29.15
29.06
25.72
22.59
14.07
O
CN
11'
5E+08
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
-5E+07
46
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

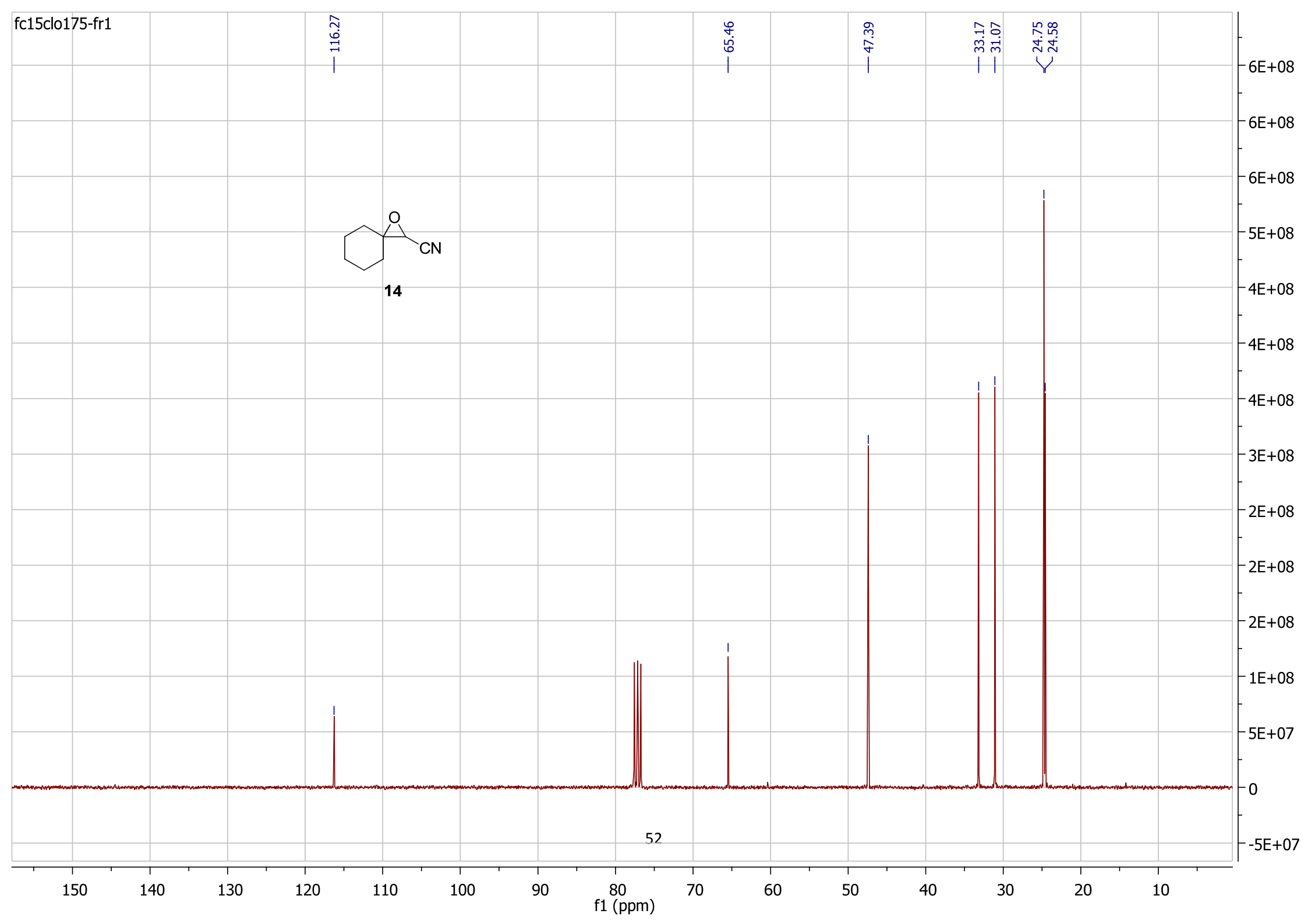

fc27clo199
3.27
1.12
1.11
1.09
1.09
1.06
1.06
0.94
0.94
0.92
0.92
0.89
0.89
O
CN
12
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
1.00
4.38
3.24
3.30
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

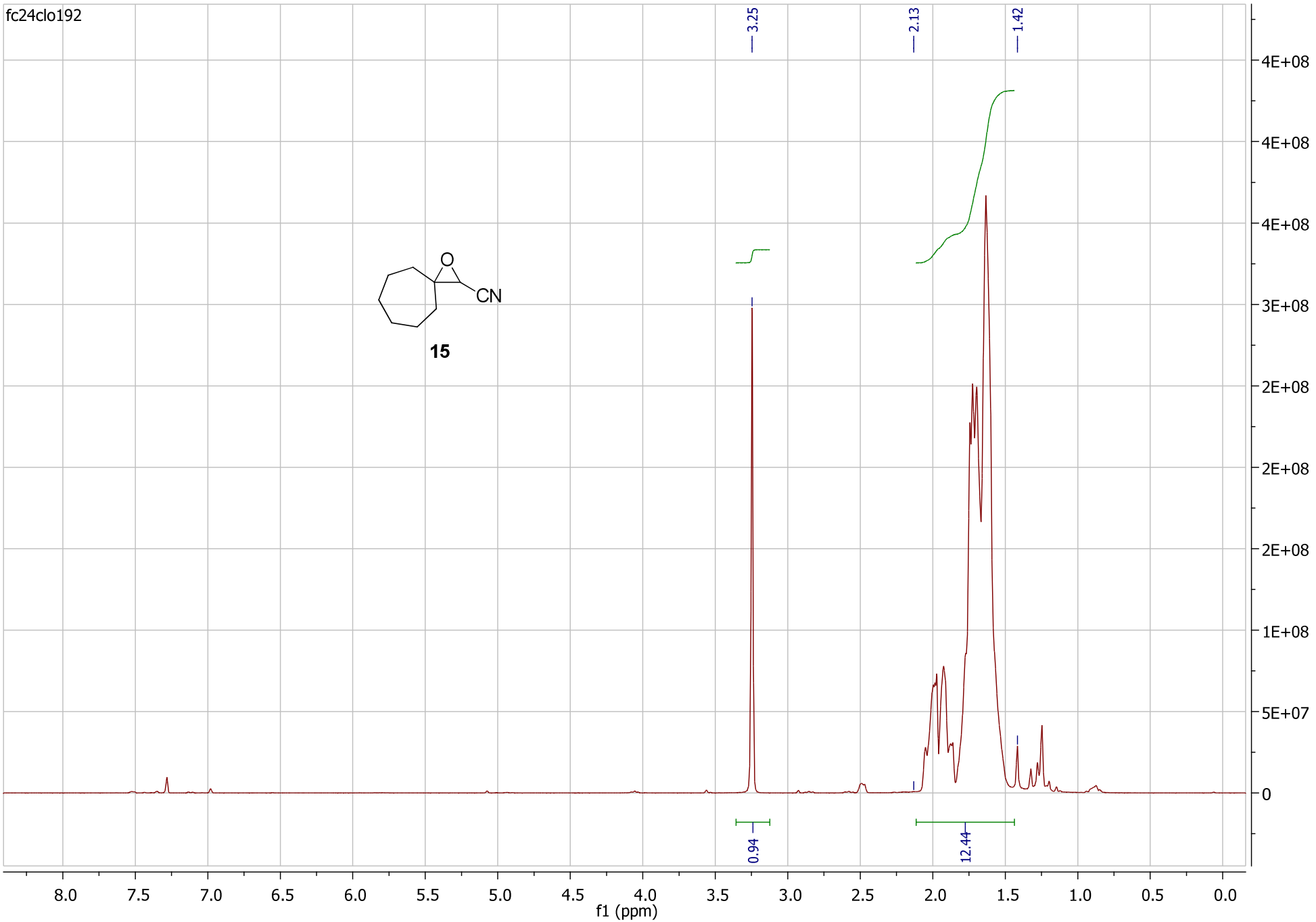

fc27clo199
116.48
67.34
46.21
25.44
25.12
9.07
8.31
O
CN
12
48
6E+08
6E+08
5E+08
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
-5E+07
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

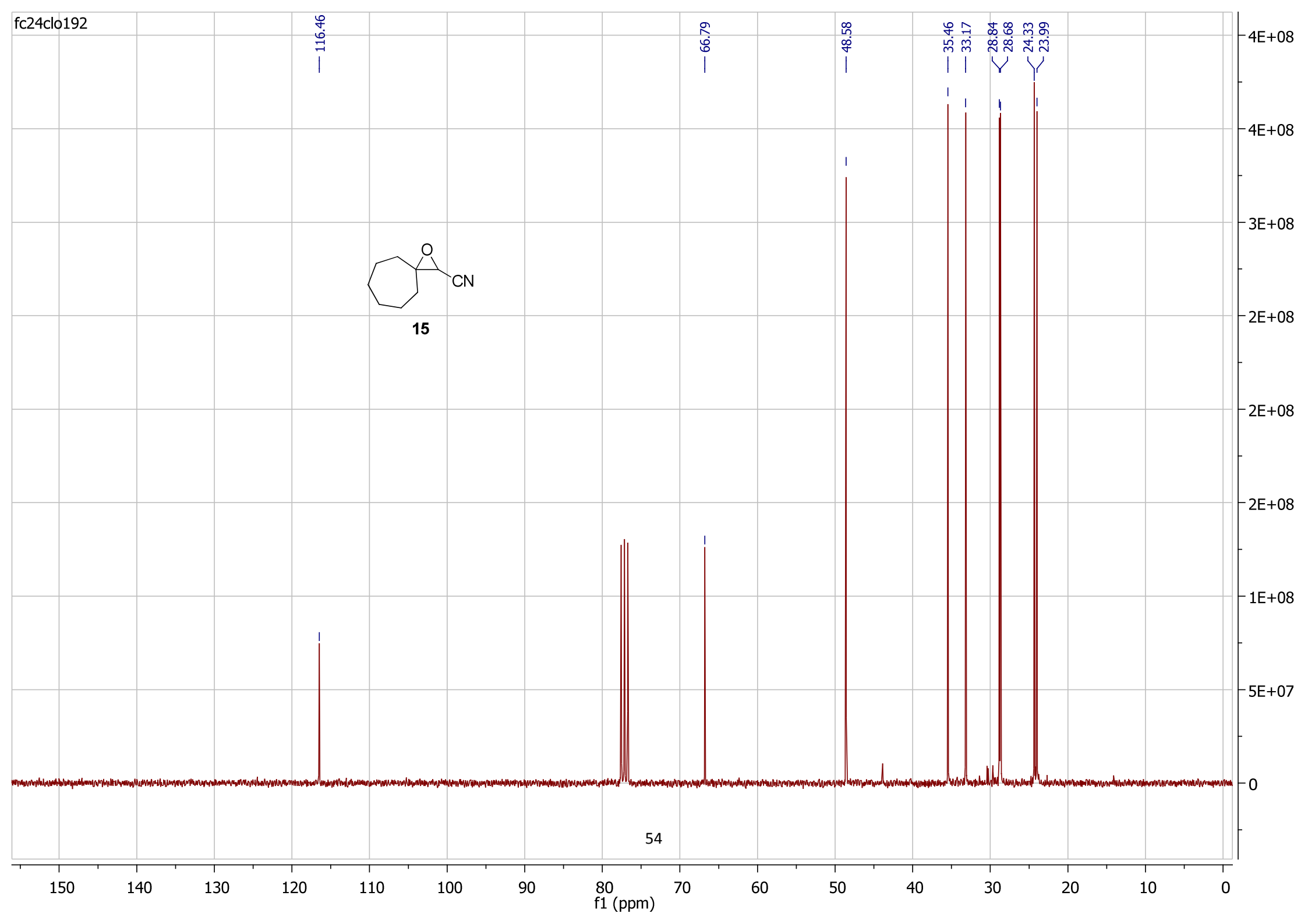

fc24clo190
3.46
2.17
1.54
O
CN
13
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
0.99
8.30
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

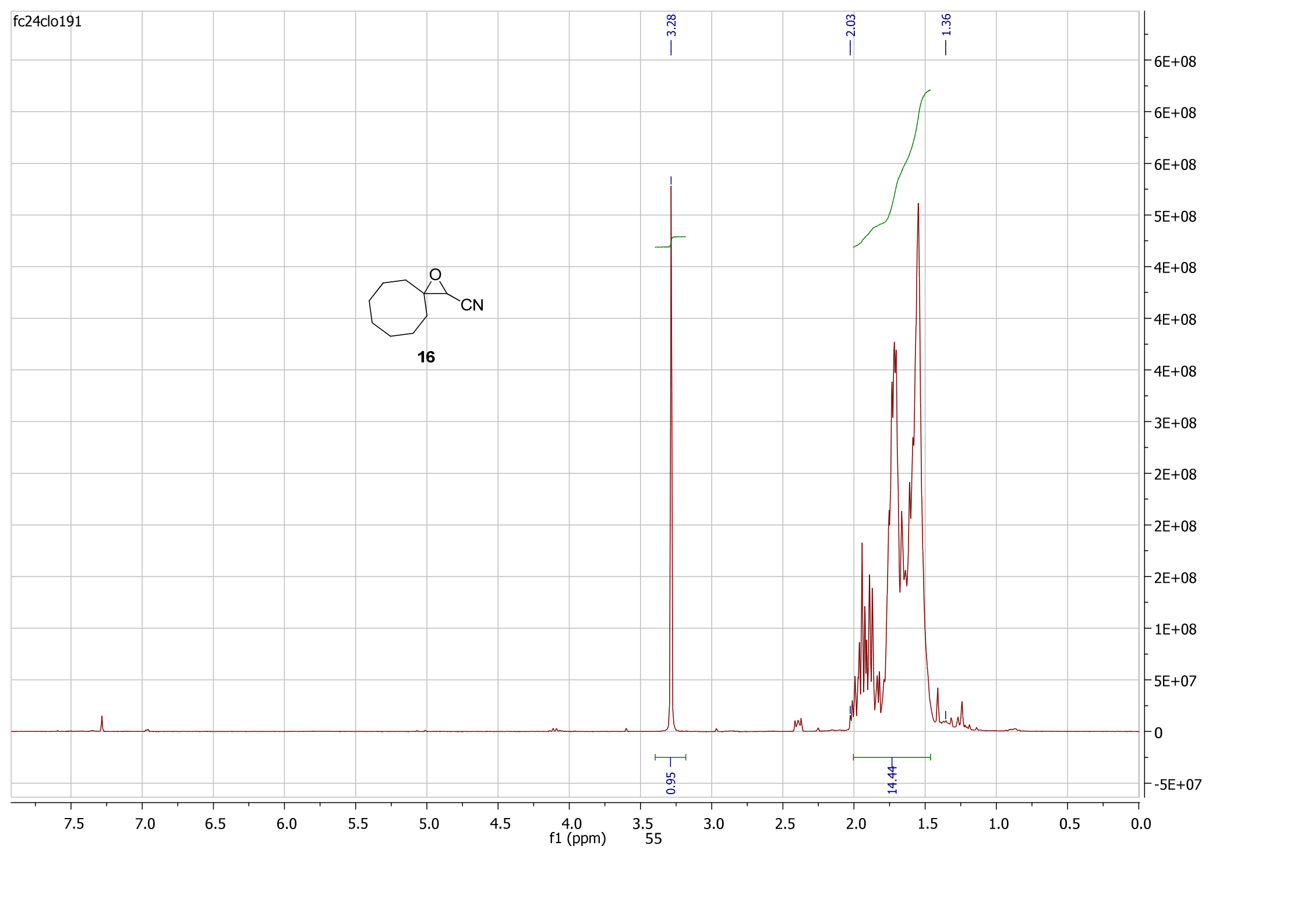

fc24clo190
116.59
71.43
45.78
31.84
30.62
25.33
24.92
O
CN
13
50
f1 (ppm)

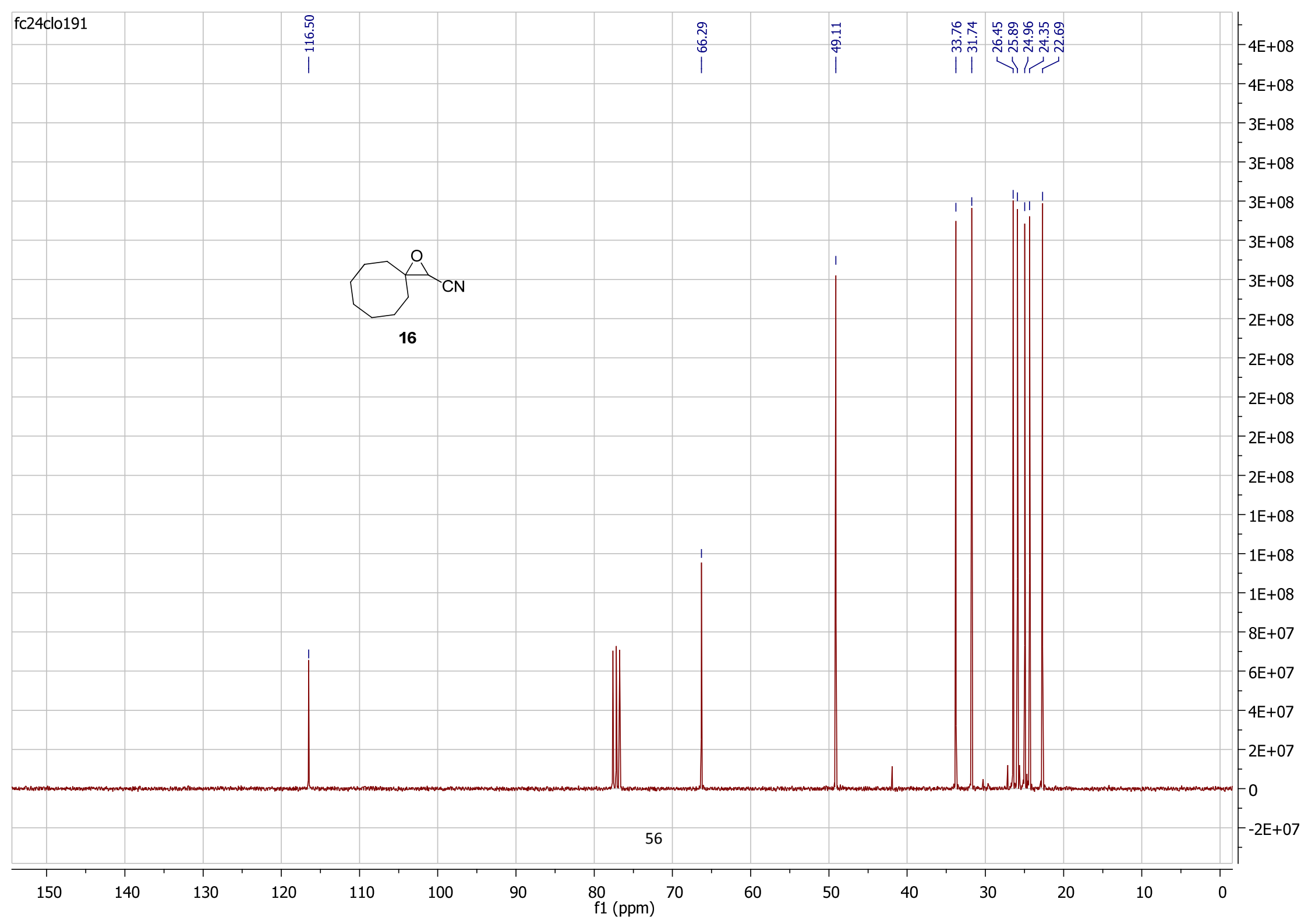

fc15clo175-fr1
3.24
1.83
1.49
O
CN
14
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
1.00
10.59
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

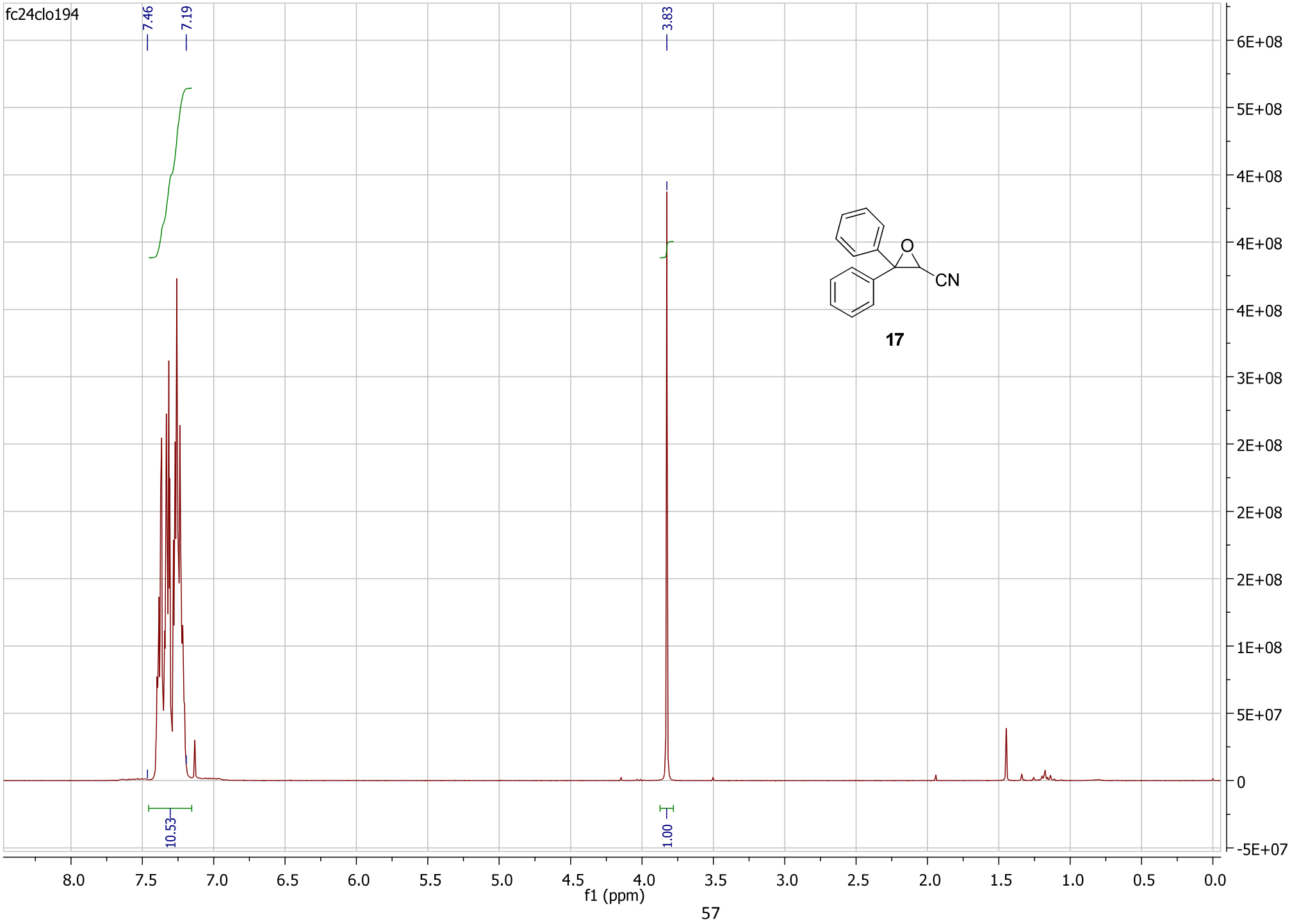

fc15clo175-fr1
116.27
65.46
47.39
33.17
31.07
24.75
24.58
O
CN
14
52
6E+08
6E+08
6E+08
5E+08
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
-5E+07
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

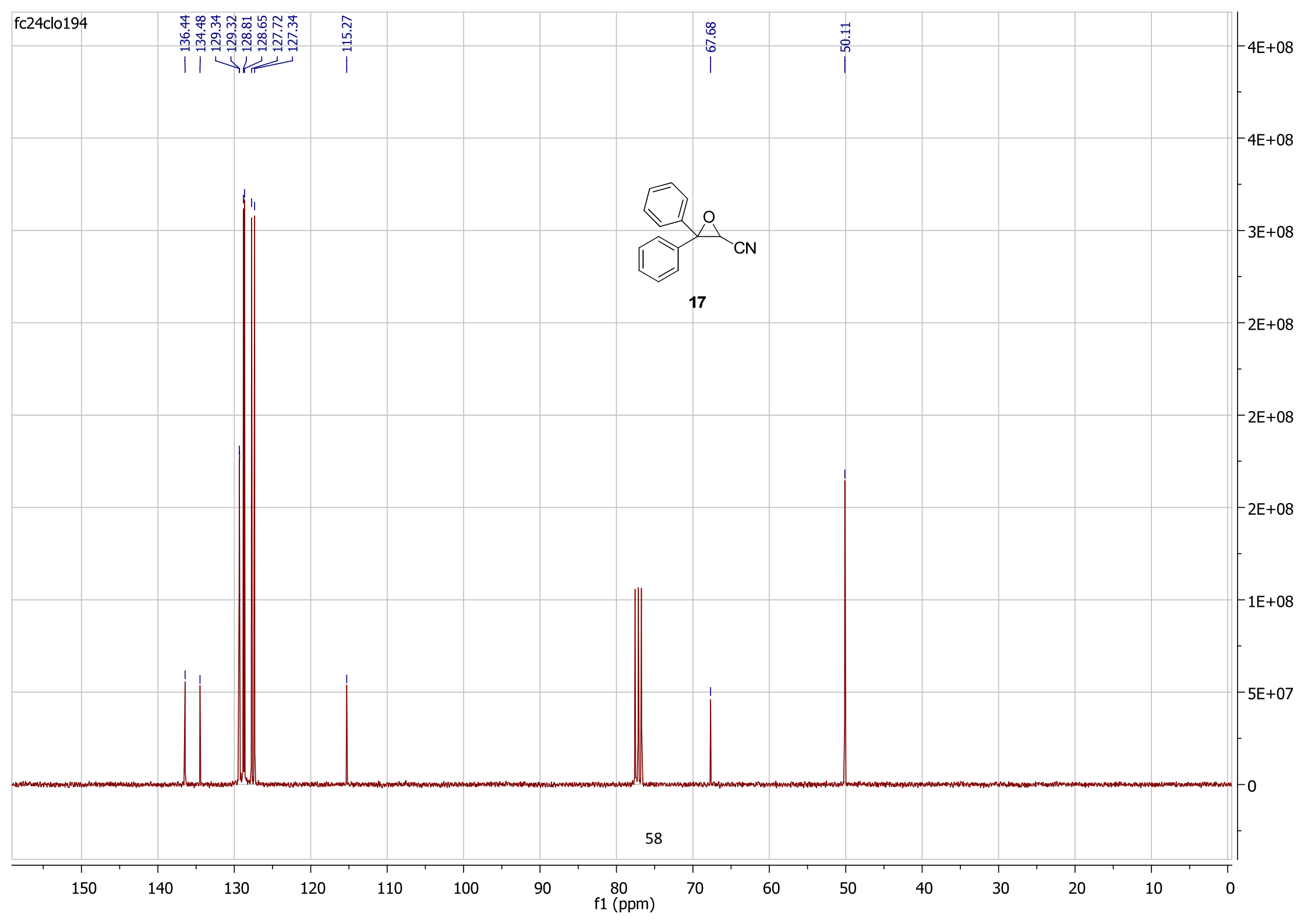

fc24clo192
3.25
2.13
1.42
O
CN
15
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
0.94
12.44
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

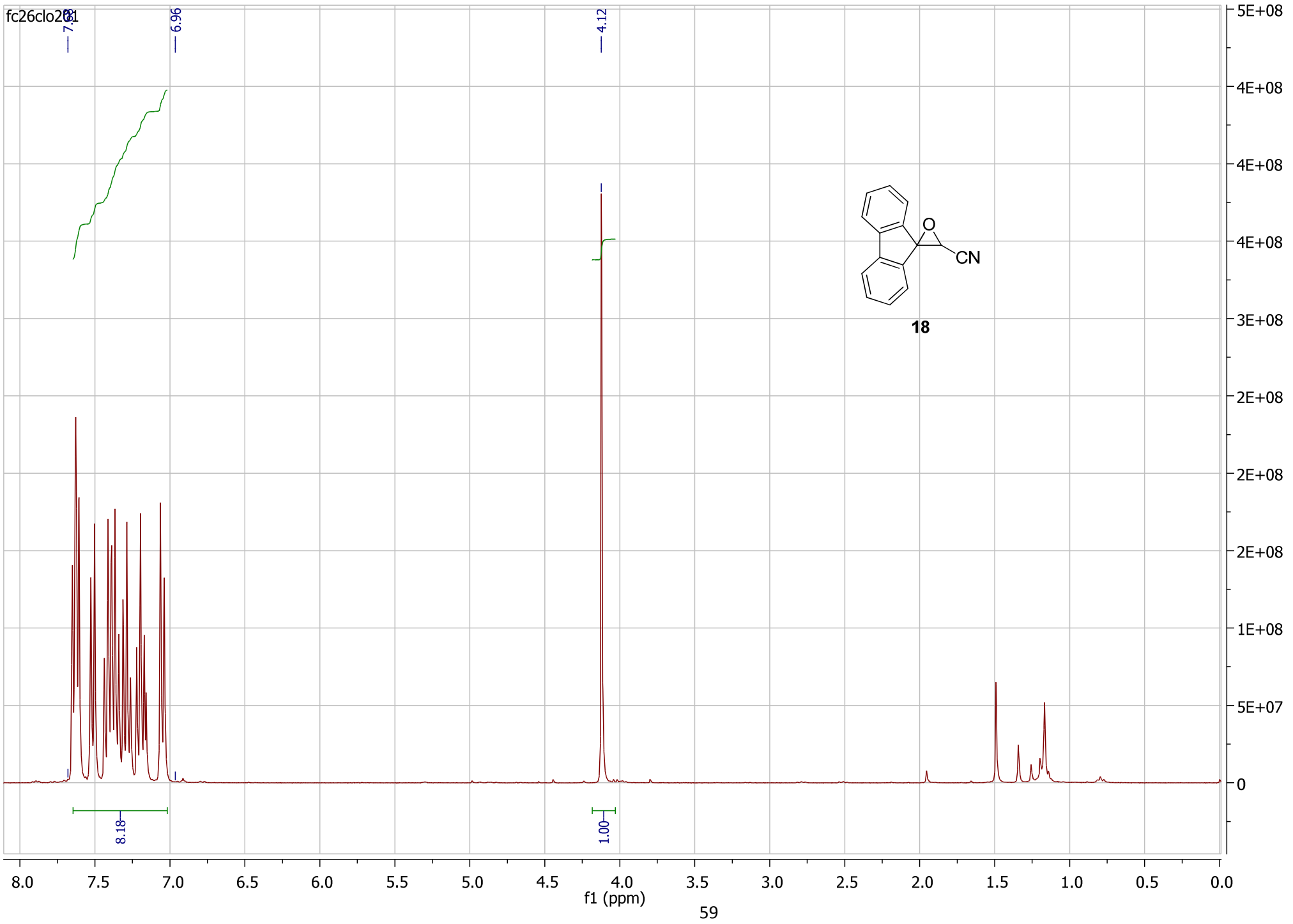

fc24clo192
116.46
66.79
48.58
35.46
33.17
28.84
28.68
24.33
23.99
O
CN
15
54
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

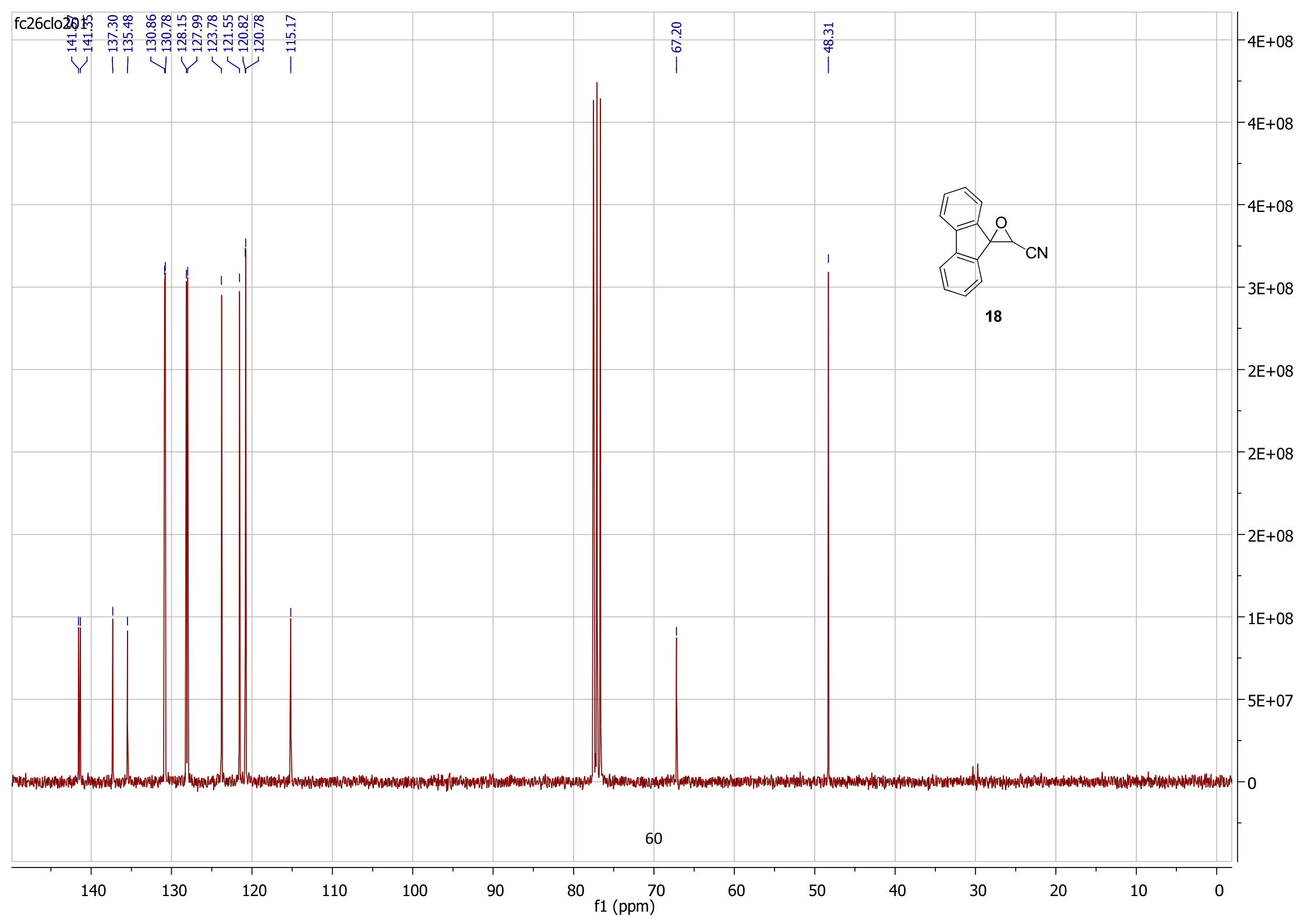

fc24clo191
3.28
2.03
1.36
O
CN
16
0.95
14.44
6E+08
6E+08
6E+08
5E+08
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
-5E+07
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
55

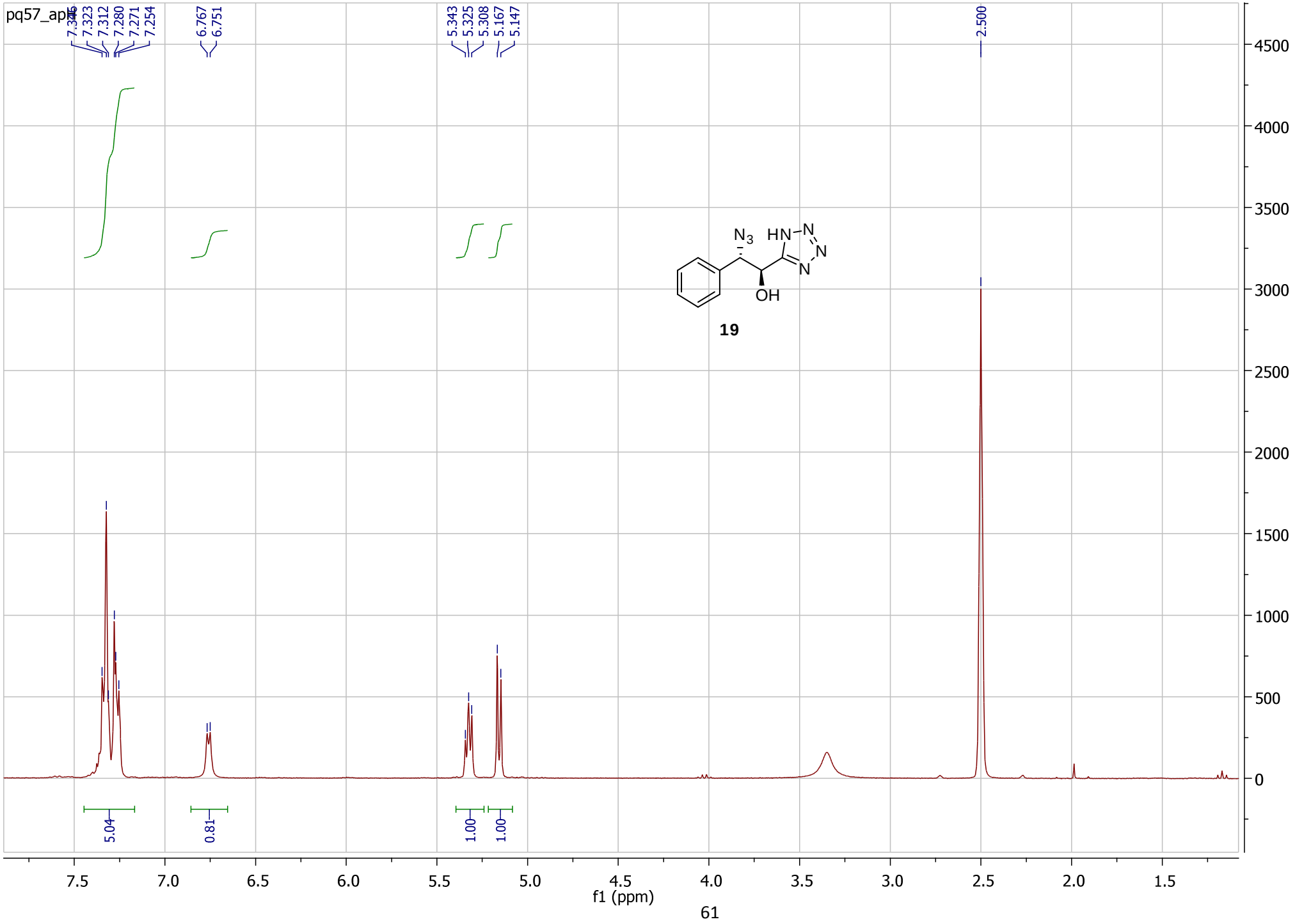

fc24clo191
116.50
66.29
49.11
33.76
31.74
26.45
25.89
24.96
24.35
22.69
O
CN
16
56
f1 (ppm)

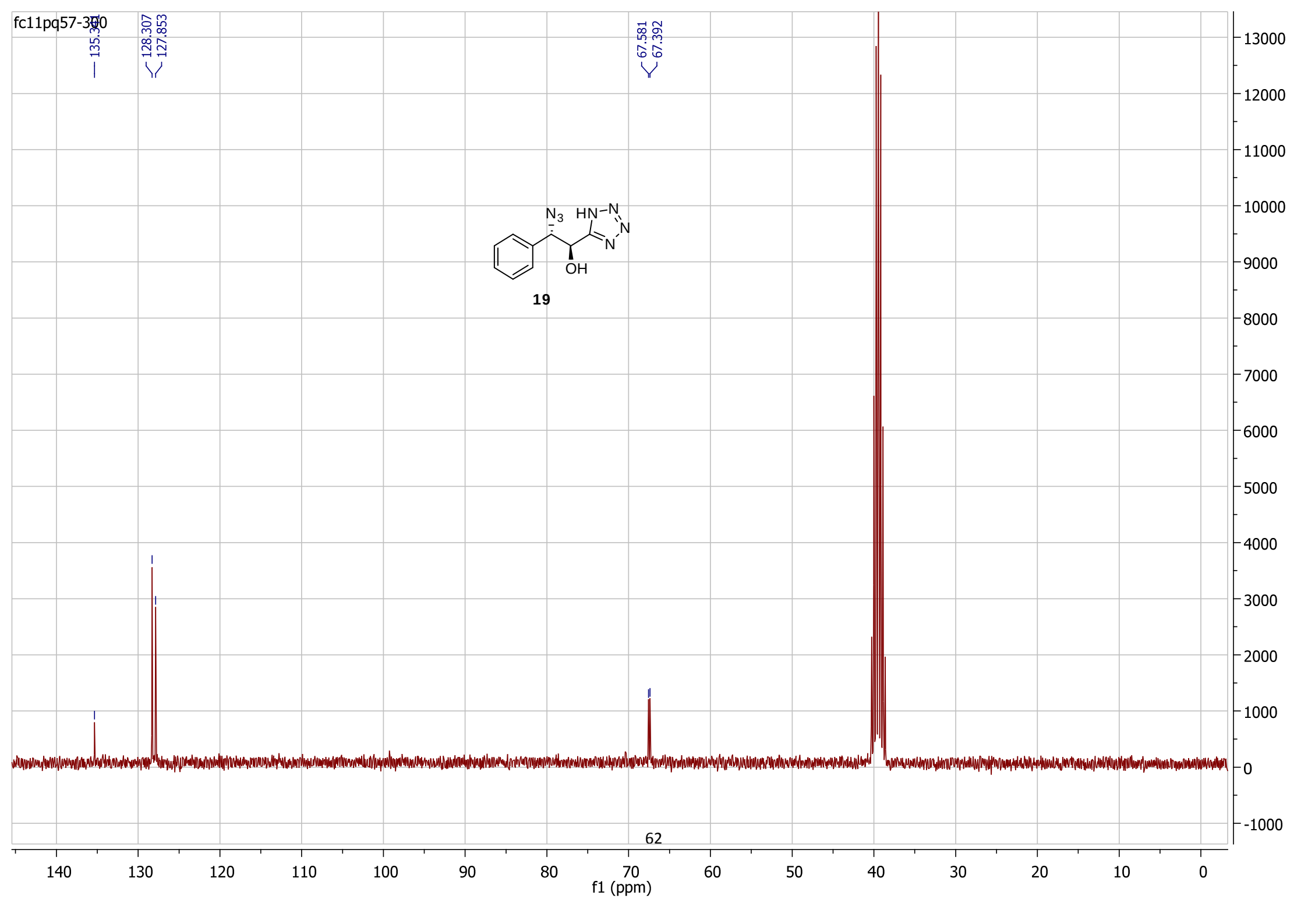

fc24clo194
7.46
7.19
3.83
O
CN
17
10.53
1.00
6E+08
5E+08
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
-5E+07
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

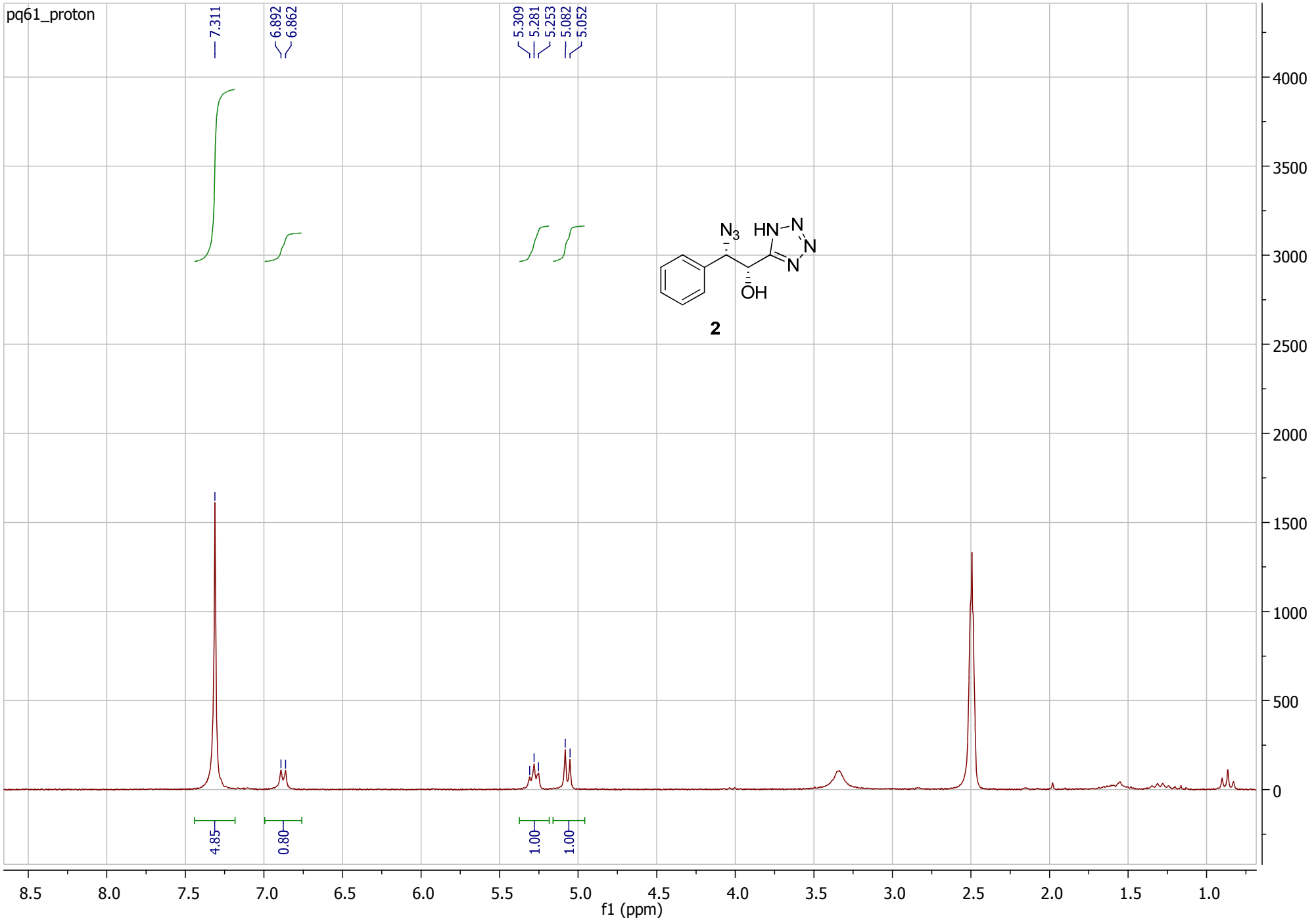

fc24clo194
136.44
134.48
129.34
129.32
128.81
128.65
127.72
127.34
115.27
67.68
50.11
O
CN
17
58
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


fc26clo2b1
7.96
6.96
4.12
O
CN
18
8.18
1.00
5E+08
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

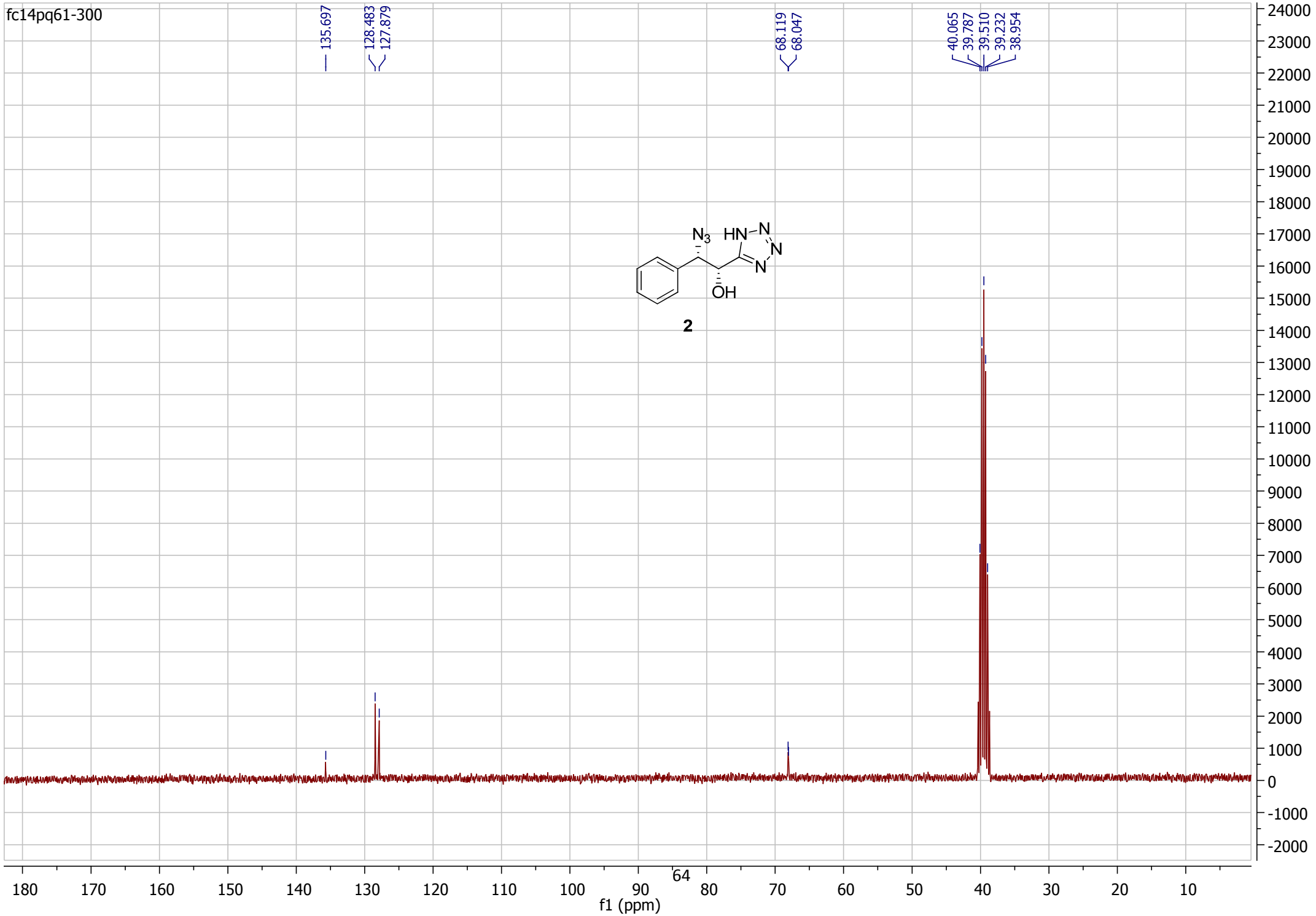

fc26clo2015
141.55
137.30
135.48
130.86
130.78
128.15
127.99
123.78
121.55
120.82
120.78
115.17
67.20
48.31
O
CN
18
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
60
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

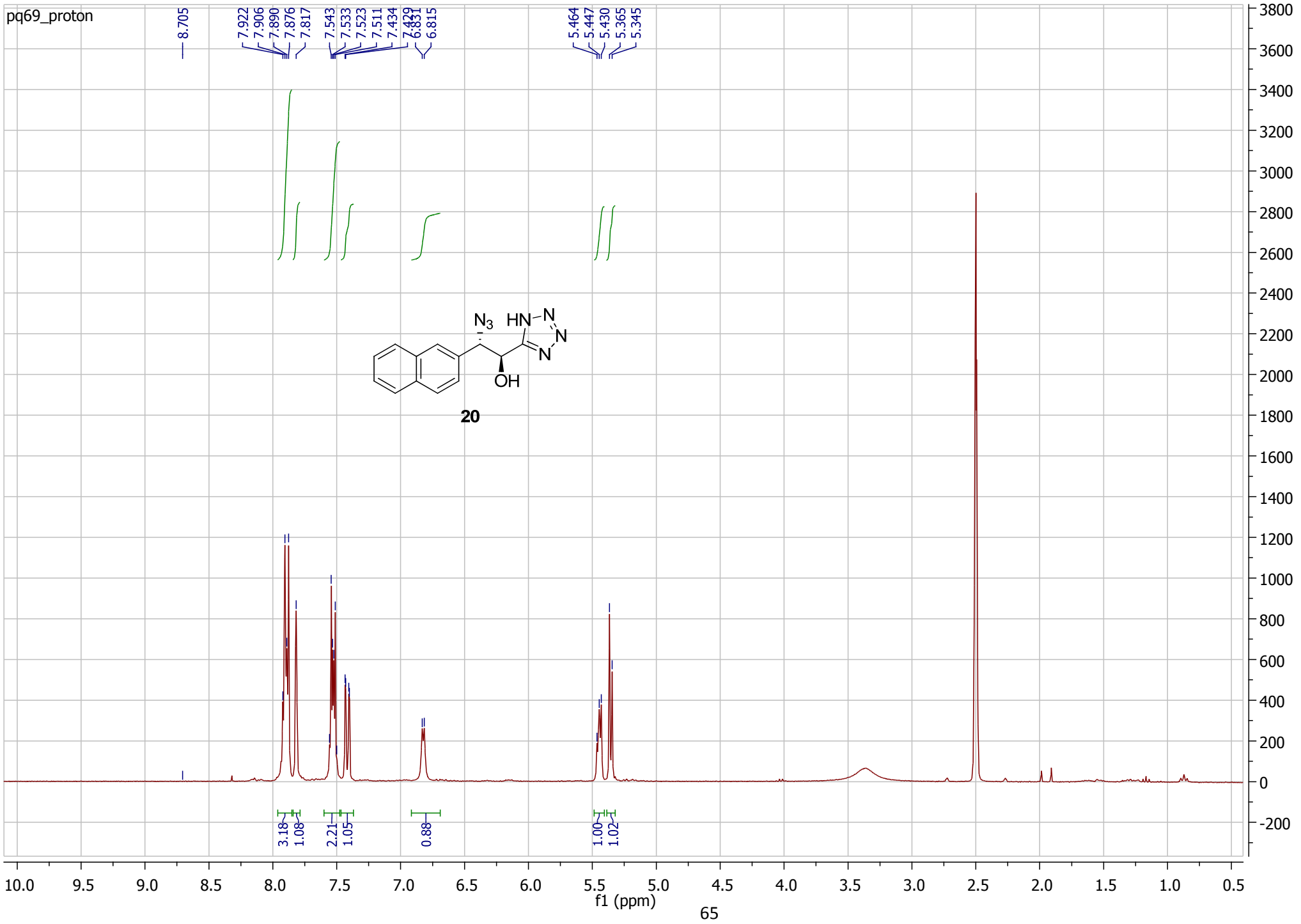

pq57_apt
7.334
7.323
7.312
7.280
7.271
7.254
6.767
6.751
5.343
5.325
5.308
5.167
5.147
2.500
N3
HN
N
N
OH
19
5.04
0.81
1.00
1.00
4500
4000
3500
3000
2500
2000
1500
1000
500
0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
f1 (ppm)

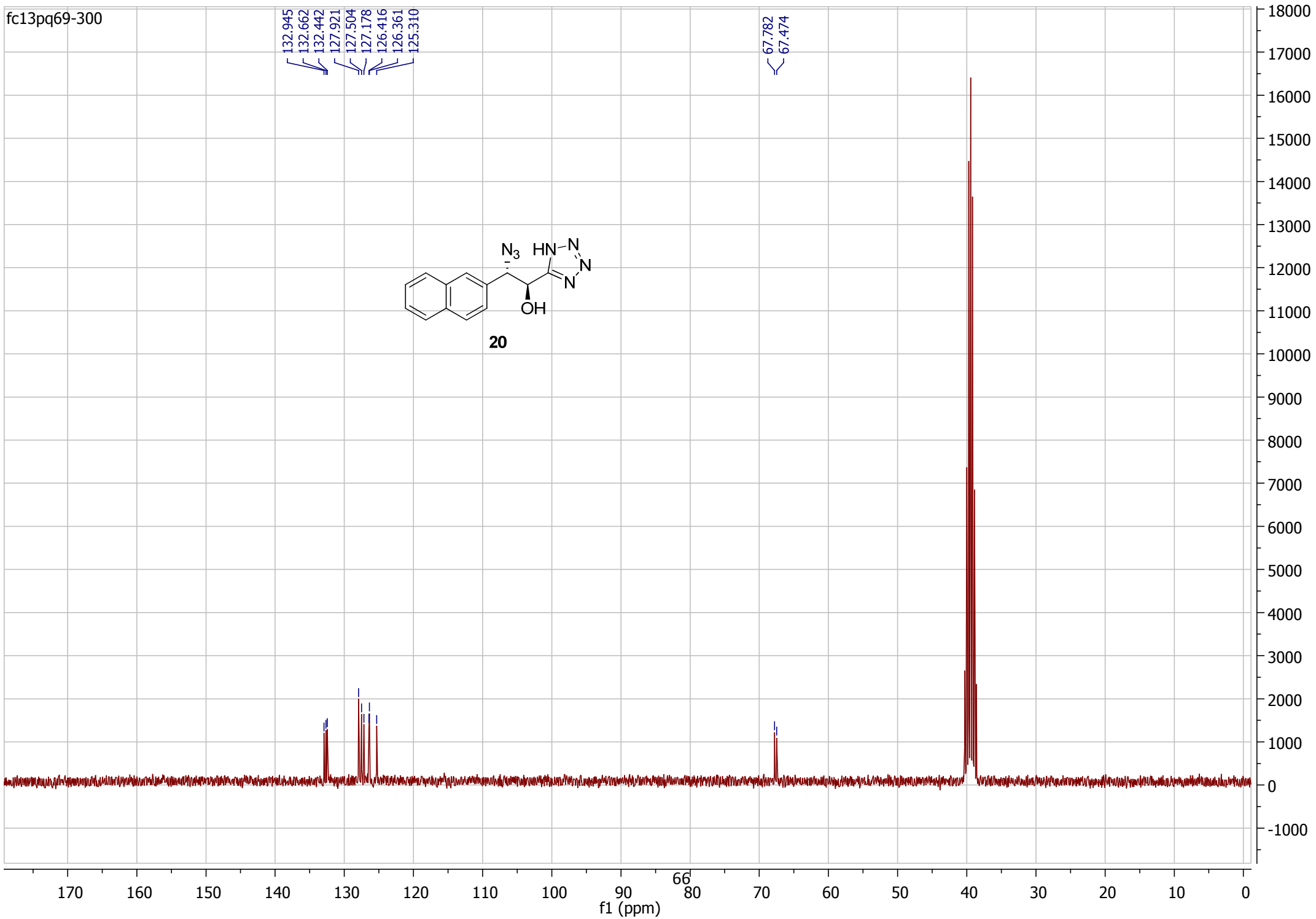

fc11pq57-300
135.340
128.307
127.853
67.581
67.392
N3
HN
N
N
N
OH
19
62
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

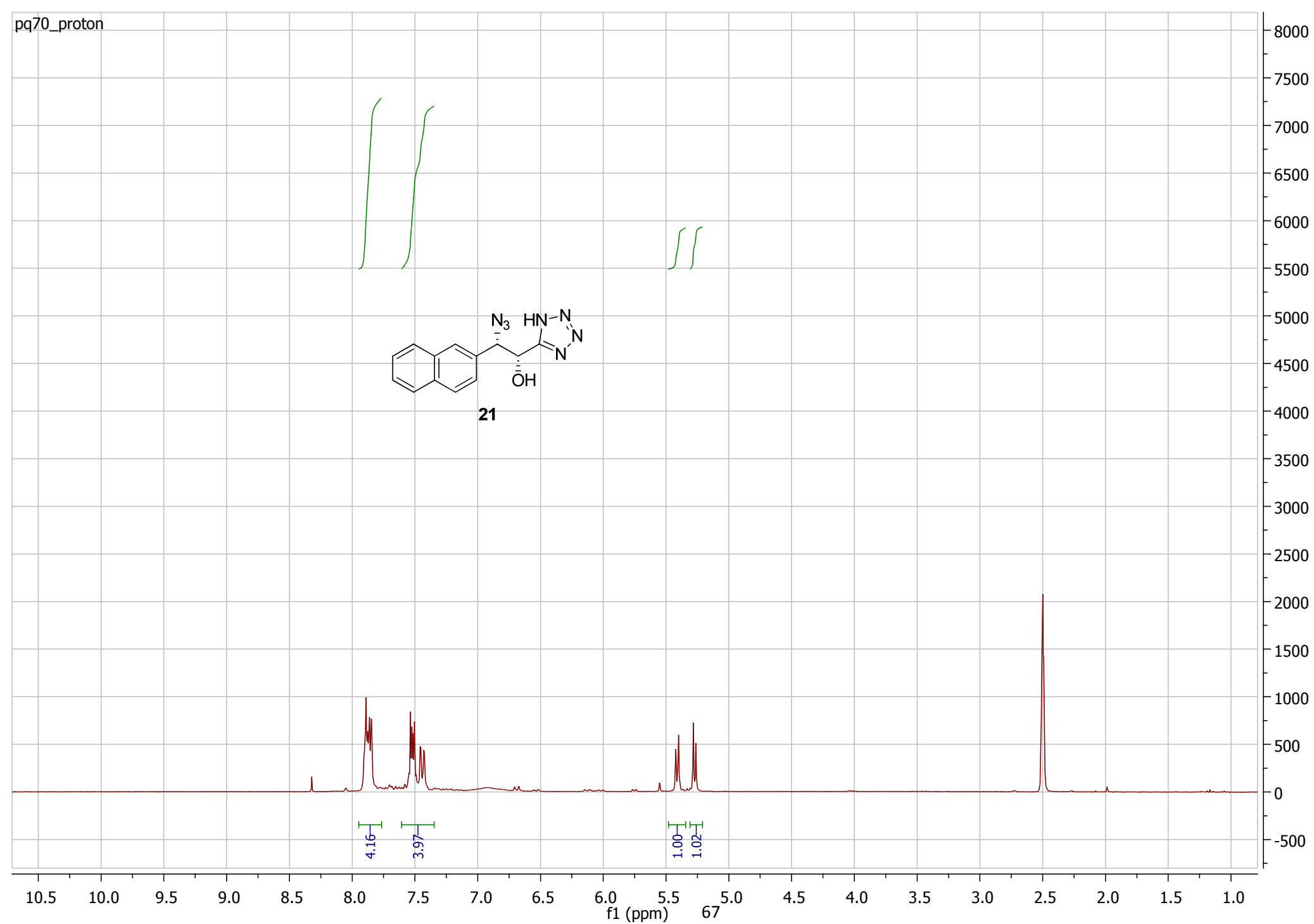

pq61_proton
7.311
6.892
6.862
5.309
5.281
5.253
5.082
5.052
4.85
0.80
1.00
1.00
f1 (ppm)

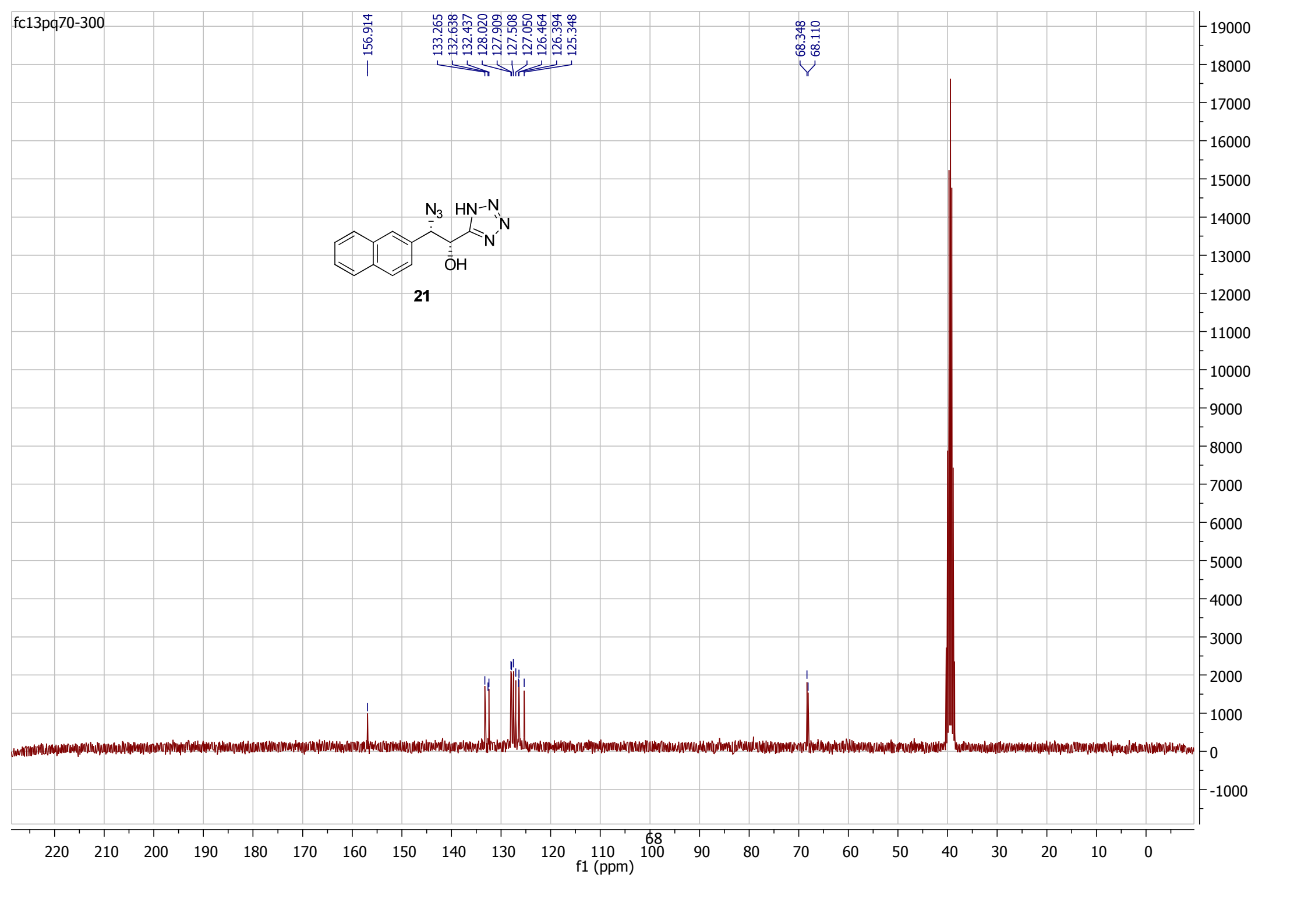
2

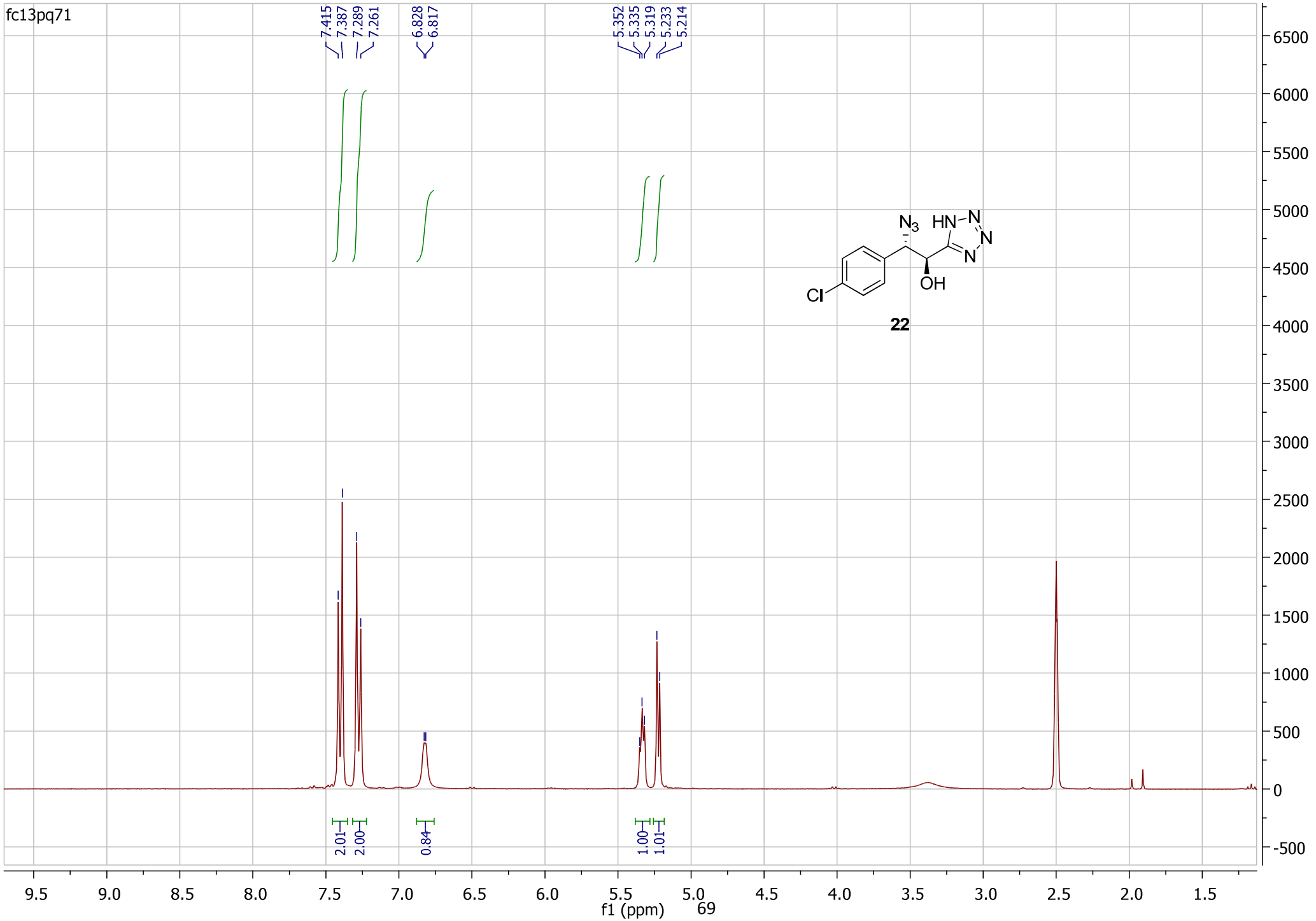

fc14pq61-300
135.697
128.483
127.879
68.119
68.047
40.065
39.787
39.510
39.232
38.954
N3
HN
N
N
N
OH
2
24000
23000
22000
21000
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

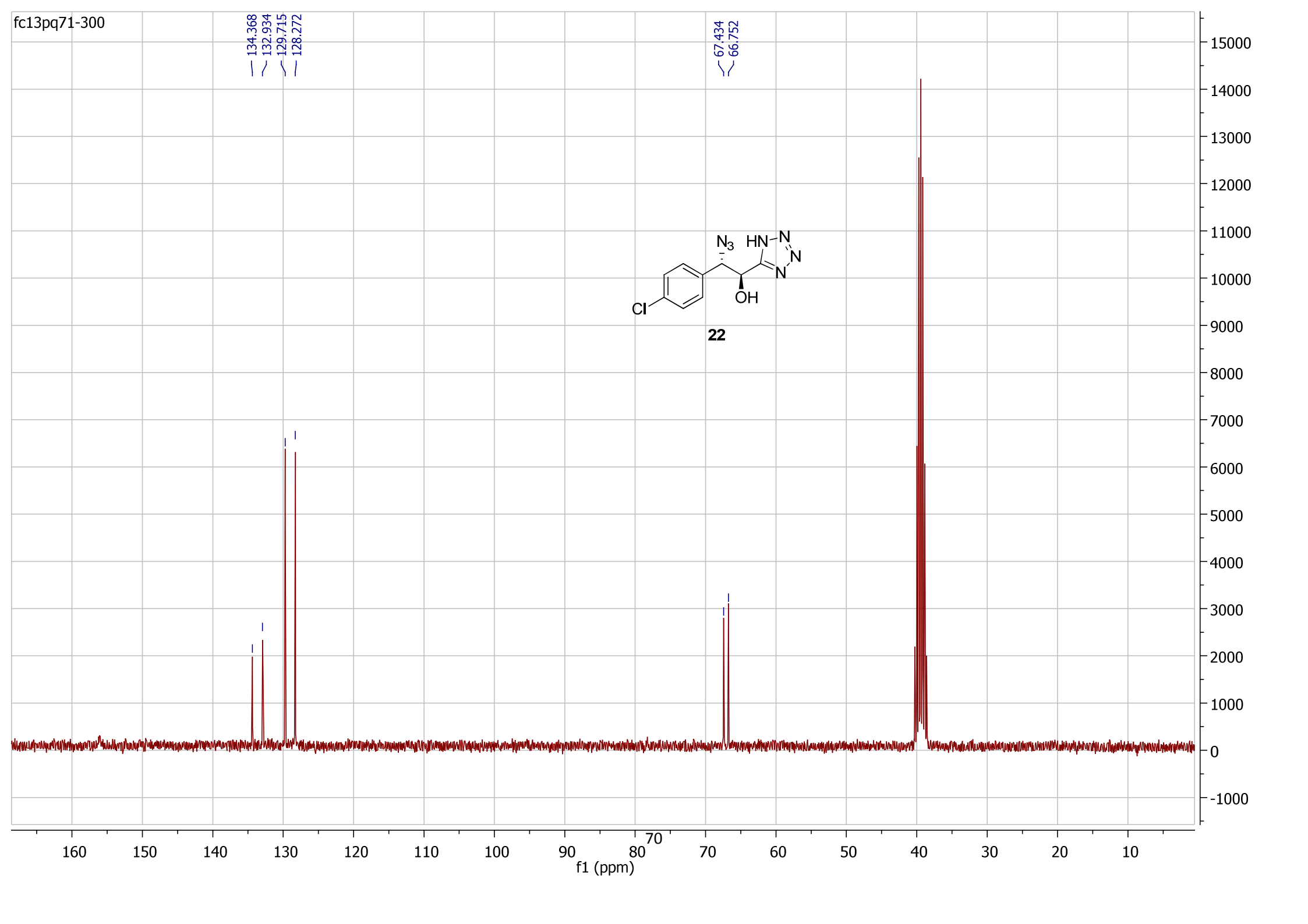

pq69_proton
8.705
7.922
7.906
7.890
7.876
7.817
7.543
7.533
7.523
7.511
7.434
7.429
6.831
6.815
5.464
5.447
5.430
5.365
5.345
N3
HN
N
N
N
OH
20
3.18
1.08
2.21
1.05
0.88
1.00
1.02
f1 (ppm)

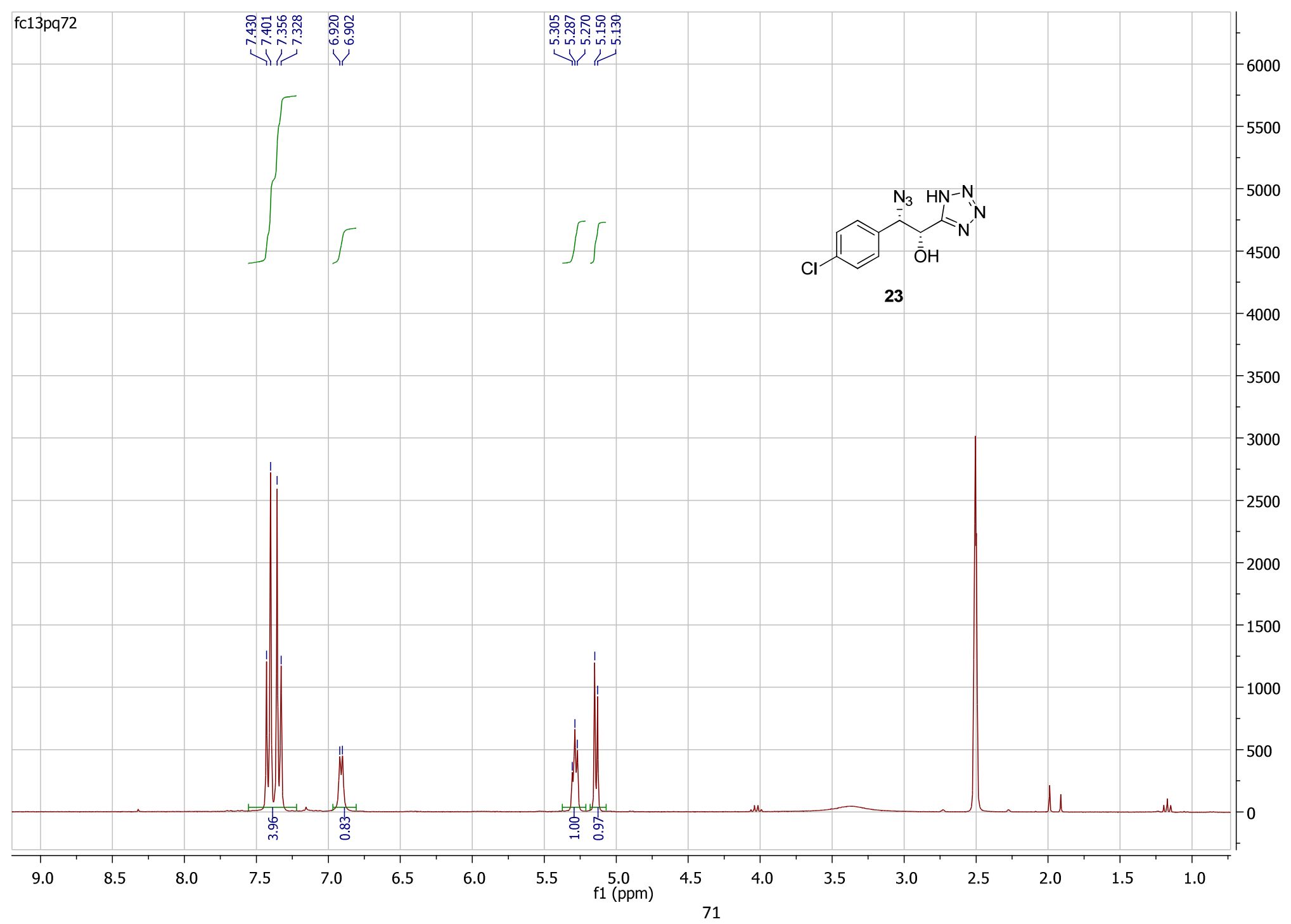

fc13pq69-300
132.945
132.662
132.442
127.921
127.504
127.178
126.416
126.361
125.310
67.782
67.474
N3
HN
N
N
N
OH
20
f1 (ppm)

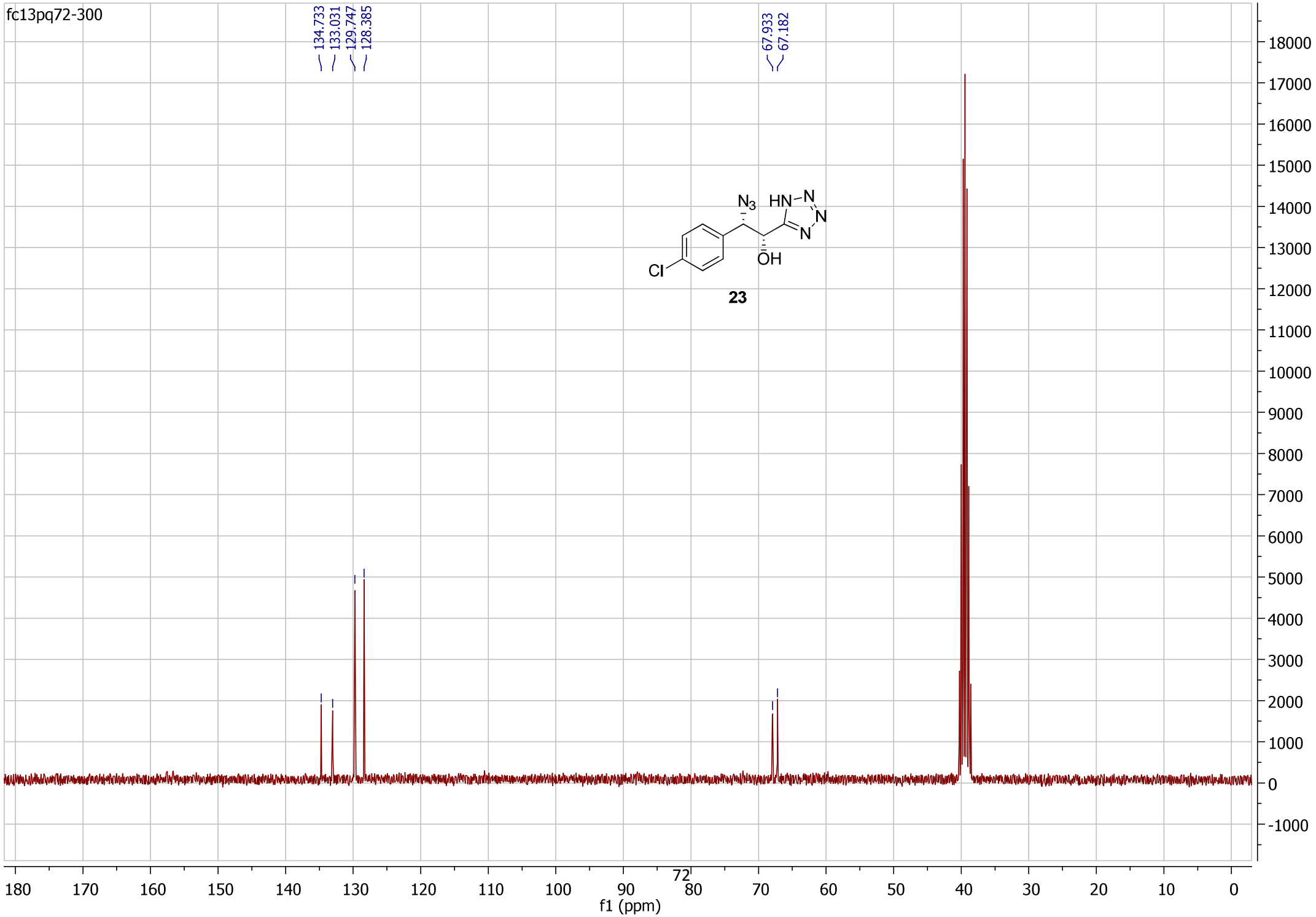

pq70_proton
N3
HN
N
N
N
OH
21
4.16
3.97
1.00
1.02
f1 (ppm)
67

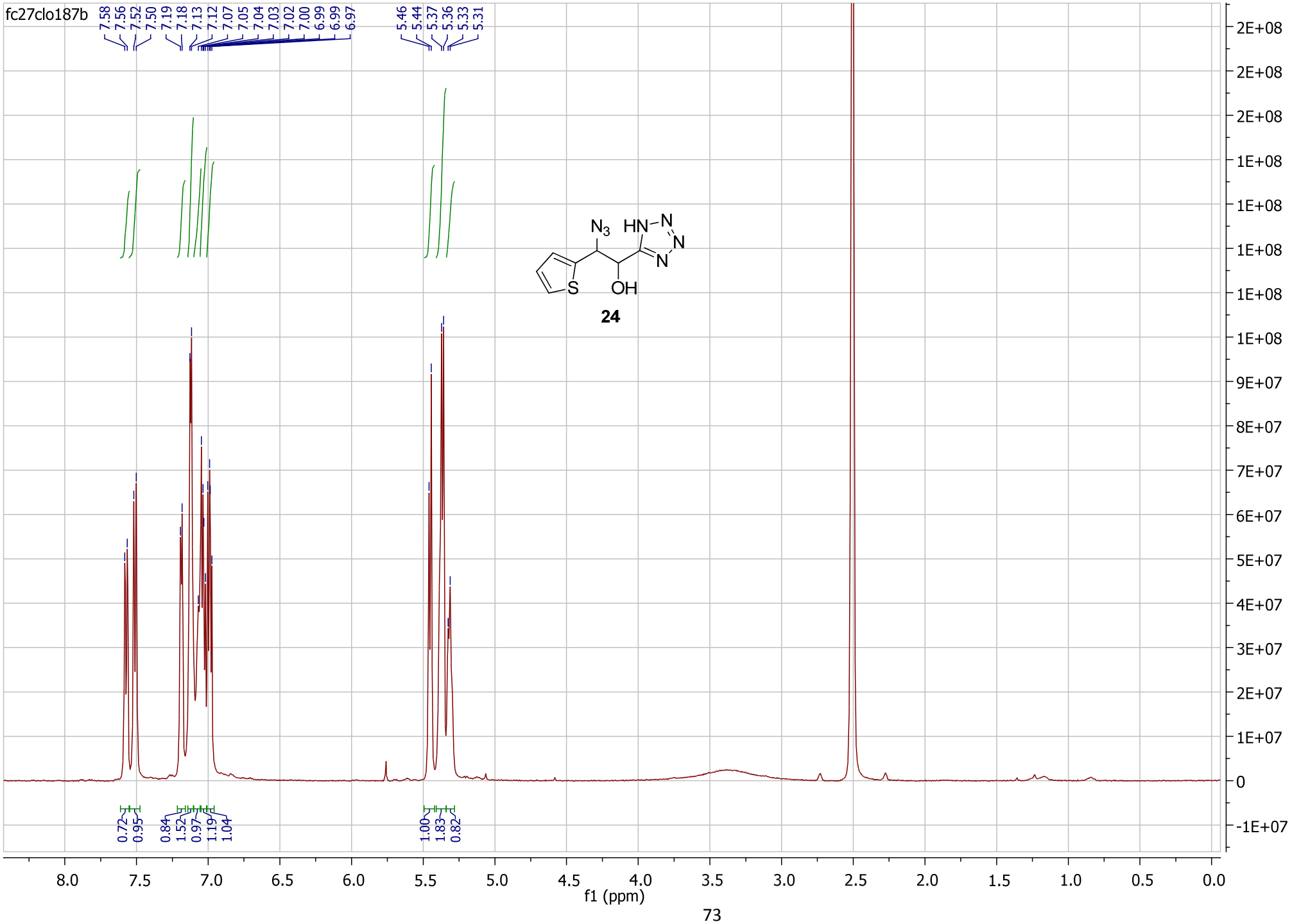

fc13pq70-300
156.914
133.265
132.638
132.437
128.020
127.909
127.508
127.050
126.464
126.394
125.348
68.348
68.110
N3
HN
N
N
N
OH
21
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

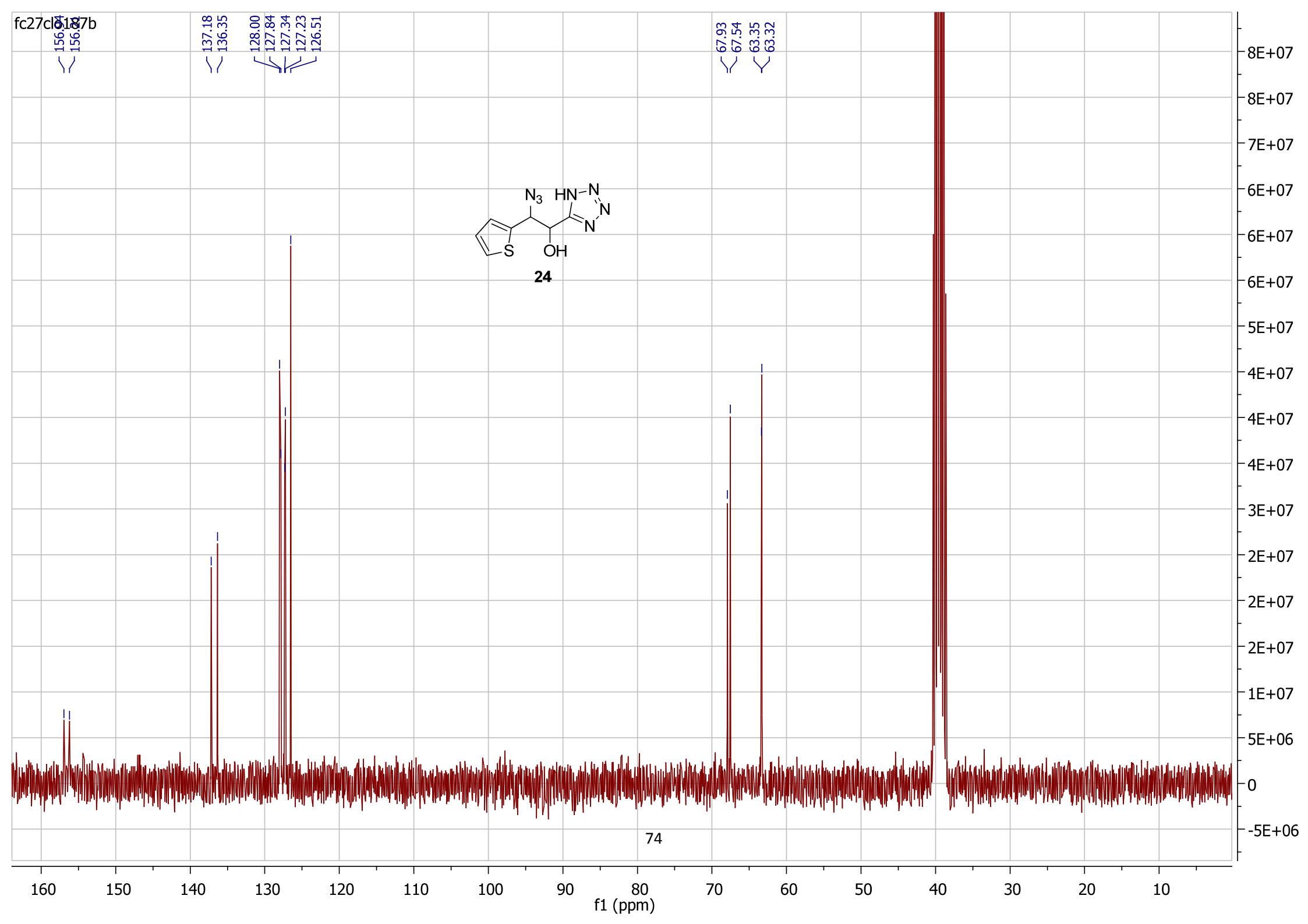

fc13pq71
7.415
7.387
7.289
7.261
6.828
6.817
5.352
5.335
5.319
5.233
5.214
N3
HN
N
N
N
OH
Cl
22
2.01
2.00
0.84
1.00
1.01
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
f1 (ppm)

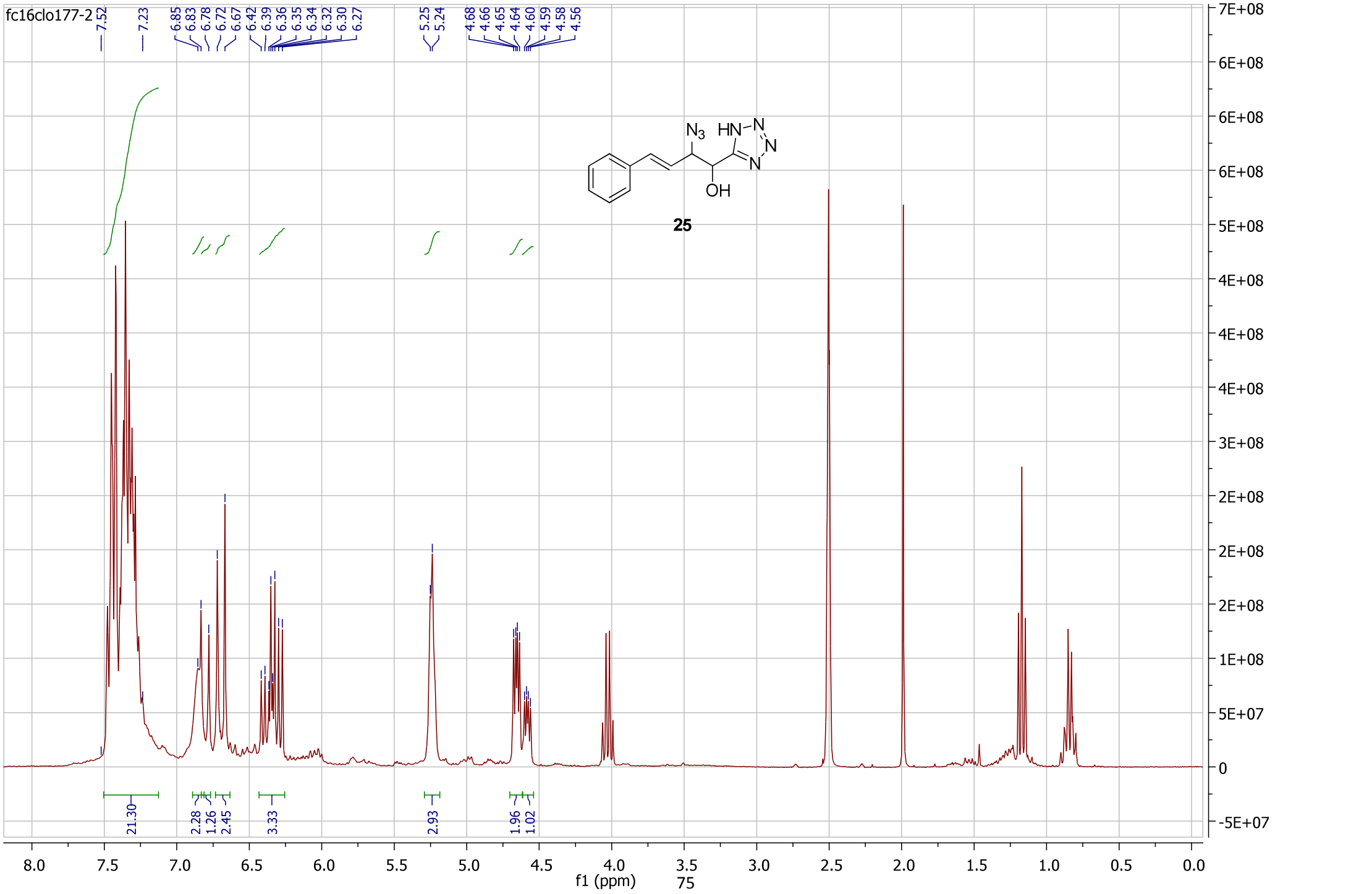

fc13pq71-300
134.368
132.934
129.715
128.272
67.434
66.752
N3
HN
N
N
N
OH
Cl
22
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

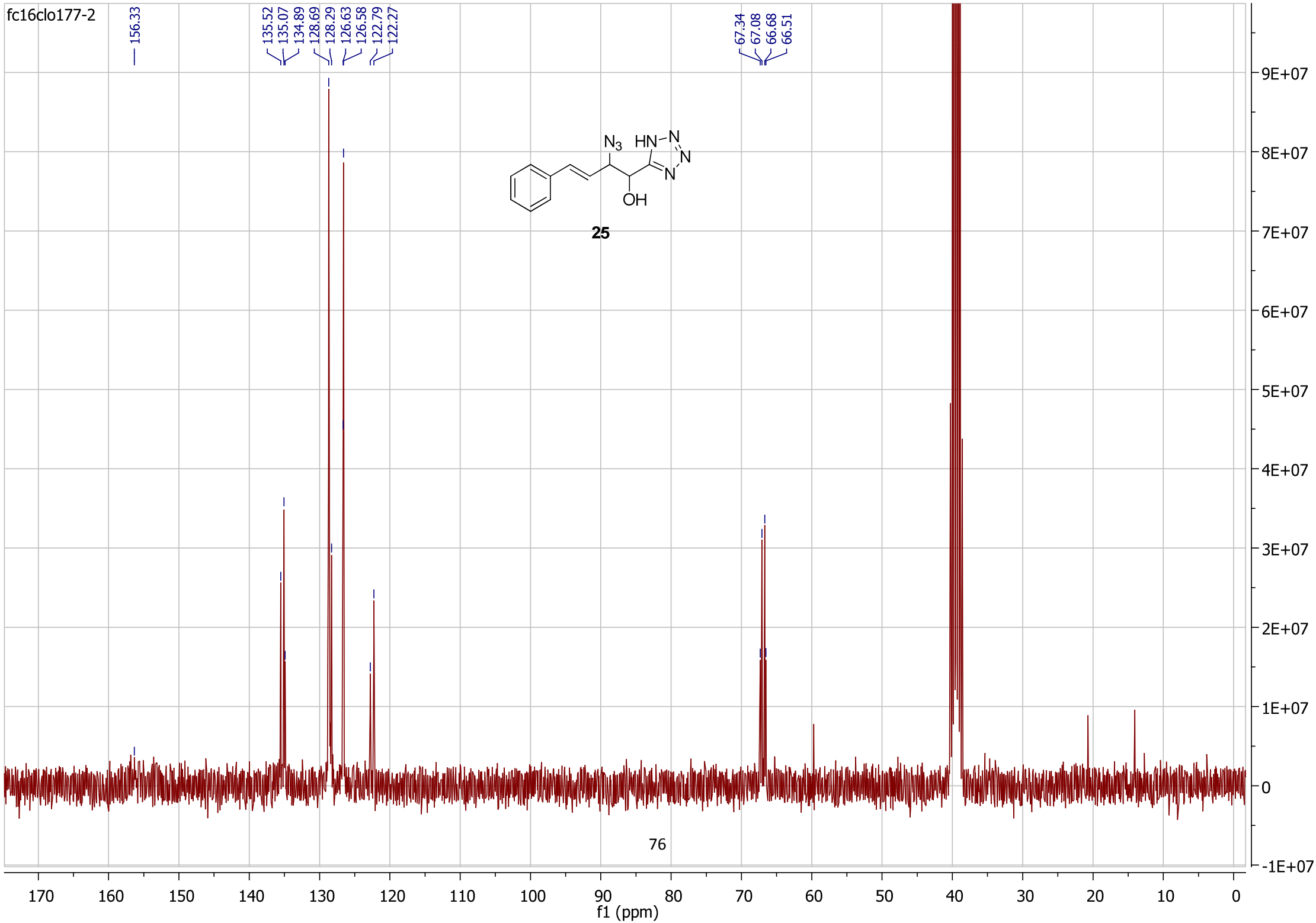

fc13pq72
7.430
7.401
7.356
7.328
6.920
6.902
5.305
5.287
5.270
5.150
5.130
N3
HN
N
N
N
OH
Cl
23
3.96
0.83
1.00
0.97
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
f1 (ppm)

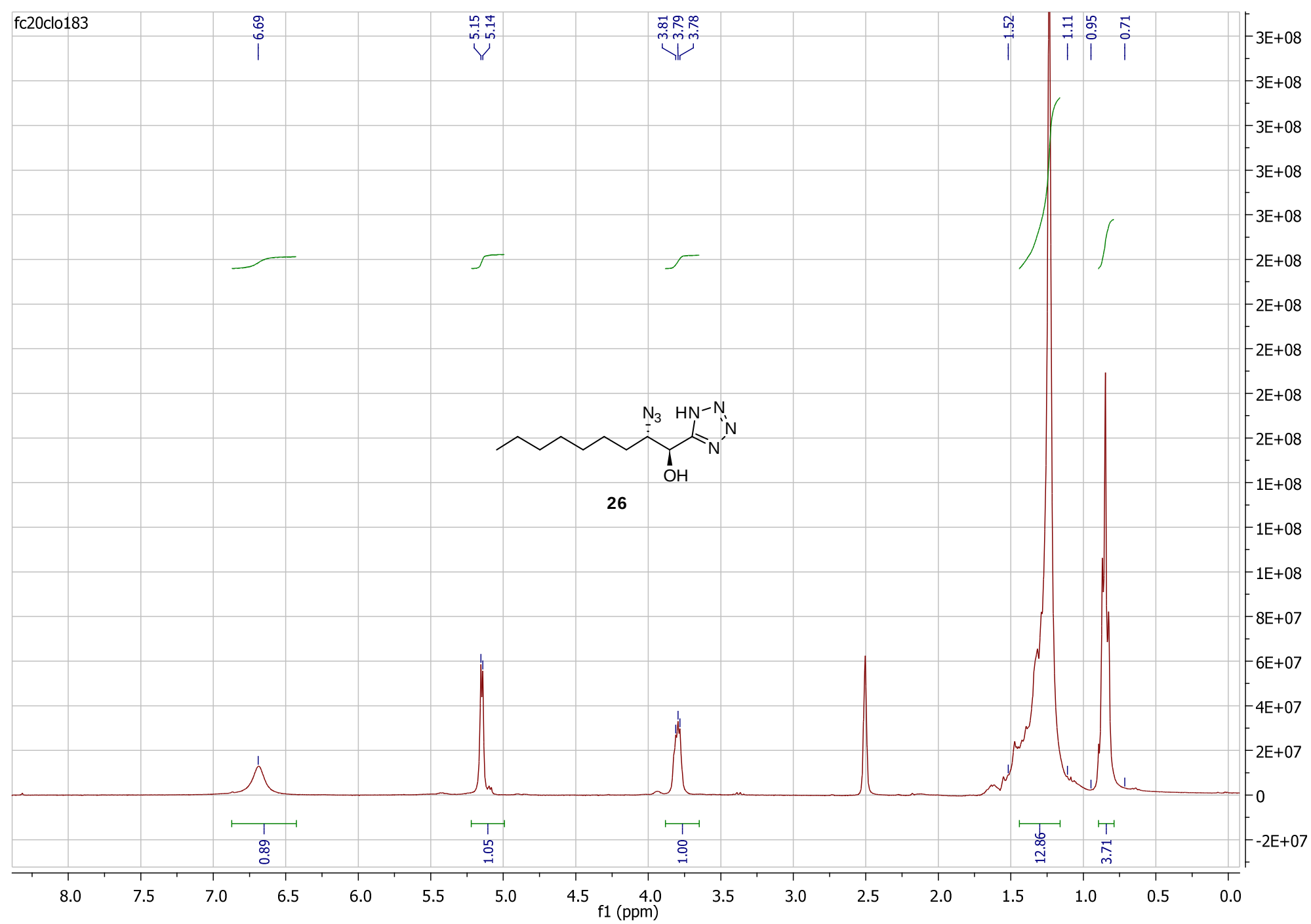

fc13pq72-300
134.733
133.031
129.747
128.385
67.933
67.182
N3
HN
N
N
N
OH
Cl
23
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

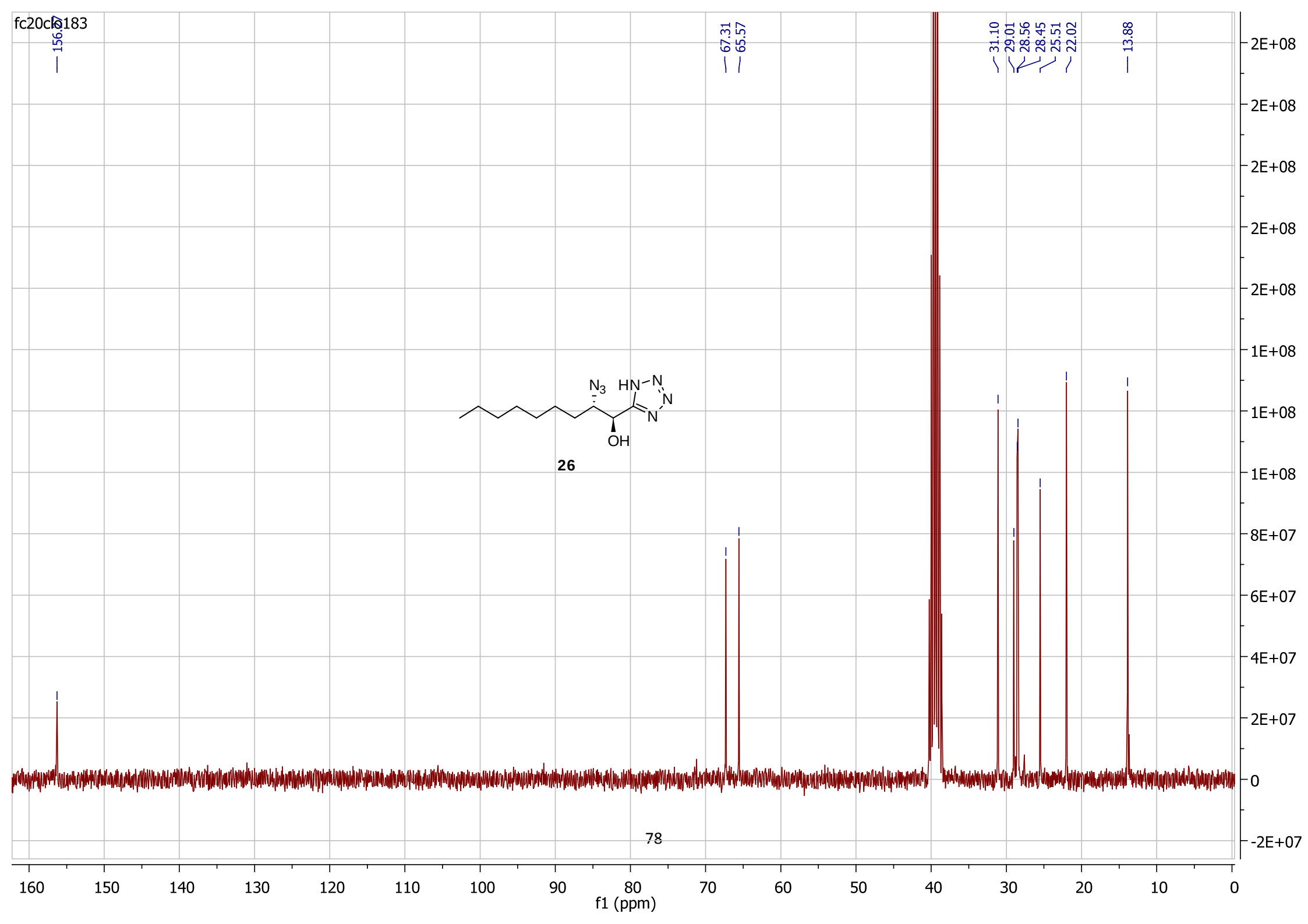

fc27clo187b
7.58
7.56
7.52
7.50
7.19
7.18
7.13
7.12
7.07
7.05
7.04
7.03
7.02
7.00
6.99
6.99
6.97
5.46
5.44
5.37
5.36
5.33
5.31
N3
HN
N
N
N
S
OH
24
0.72
0.95
0.84
1.52
0.97
1.19
1.04
1.00
1.83
0.82
2E+08
2E+08
2E+08
1E+08
1E+08
1E+08
1E+08
1E+08
1E+08
9E+07
8E+07
7E+07
6E+07
5E+07
4E+07
3E+07
2E+07
1E+07
0
-1E+07
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

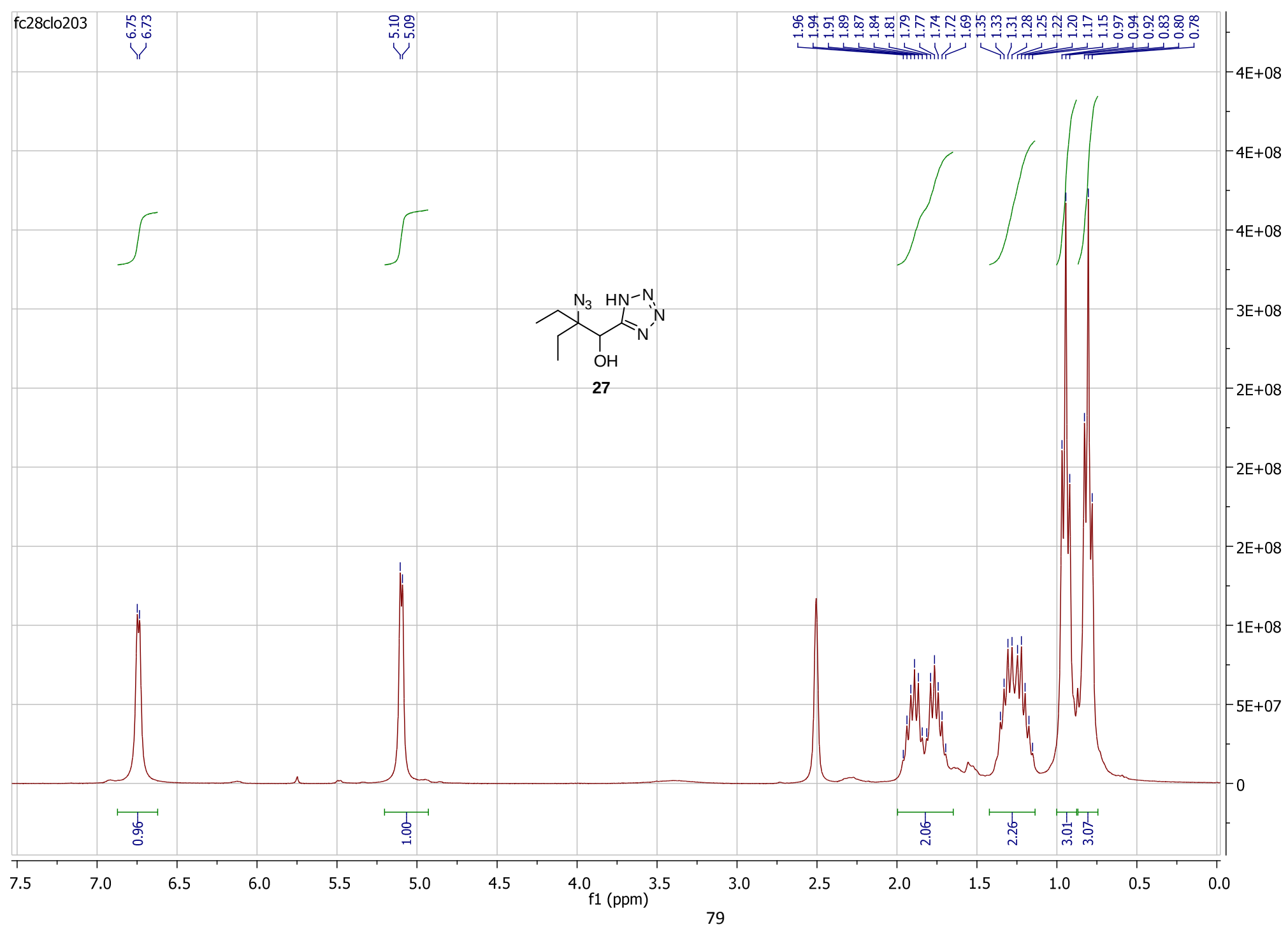

fc27cl0187b
156.54
156.14
137.18
136.35
128.00
127.84
127.34
127.23
126.51
67.93
67.54
63.35
63.32
N3
HN
N
N
N
S
OH
24
74
f1 (ppm)

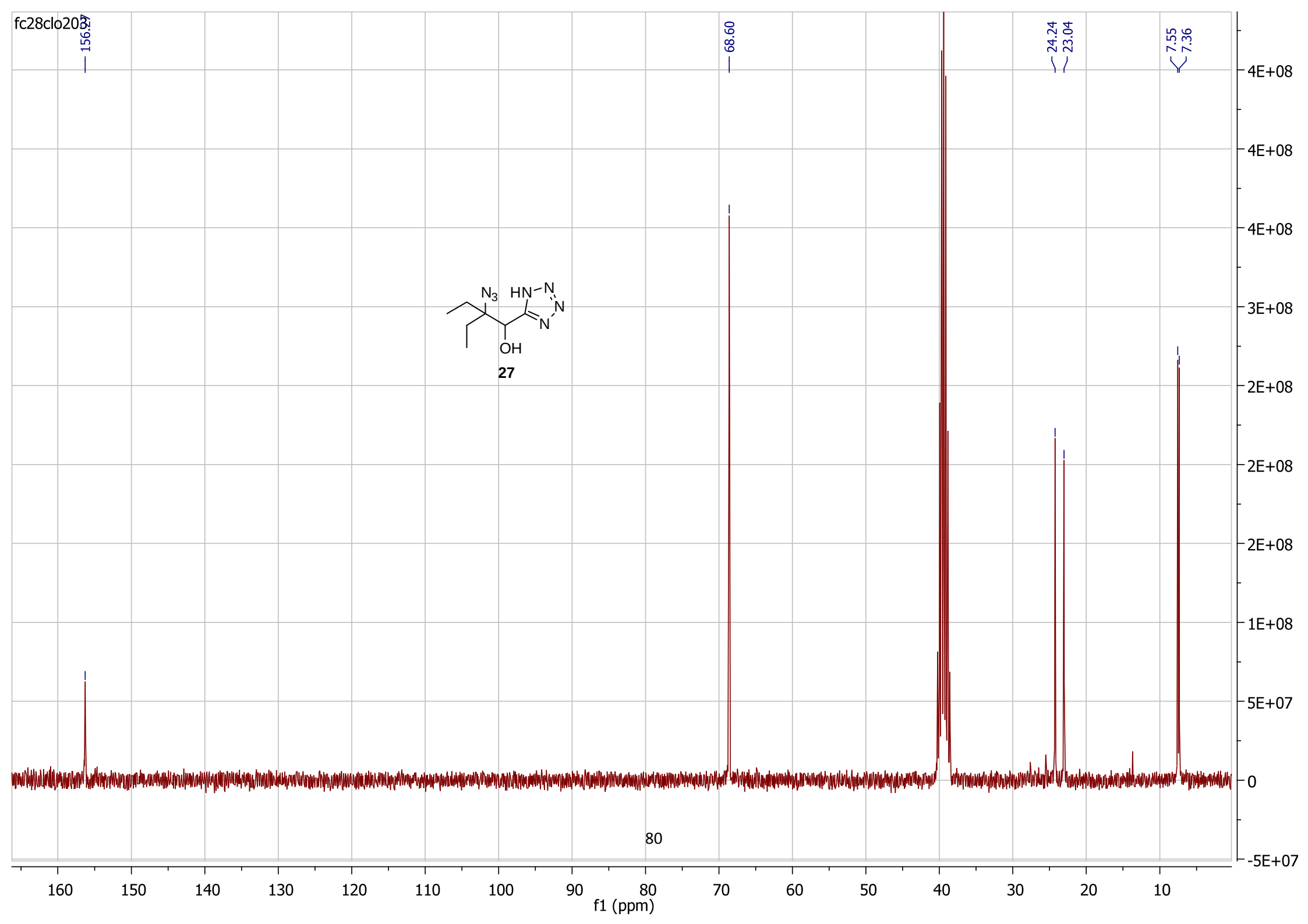

fc16clo177-2
7.52
7.23
6.85
6.83
6.78
6.72
6.67
6.42
6.39
6.36
6.35
6.34
6.32
6.30
6.27
5.25
5.24
4.68
4.66
4.65
4.64
4.60
4.59
4.58
4.56
N3
HN
N
N
N
OH
25
21.30
2.28
1.26
2.45
3.33
2.93
1.96
1.02
7E+08
6E+08
6E+08
6E+08
5E+08
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
-5E+07
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

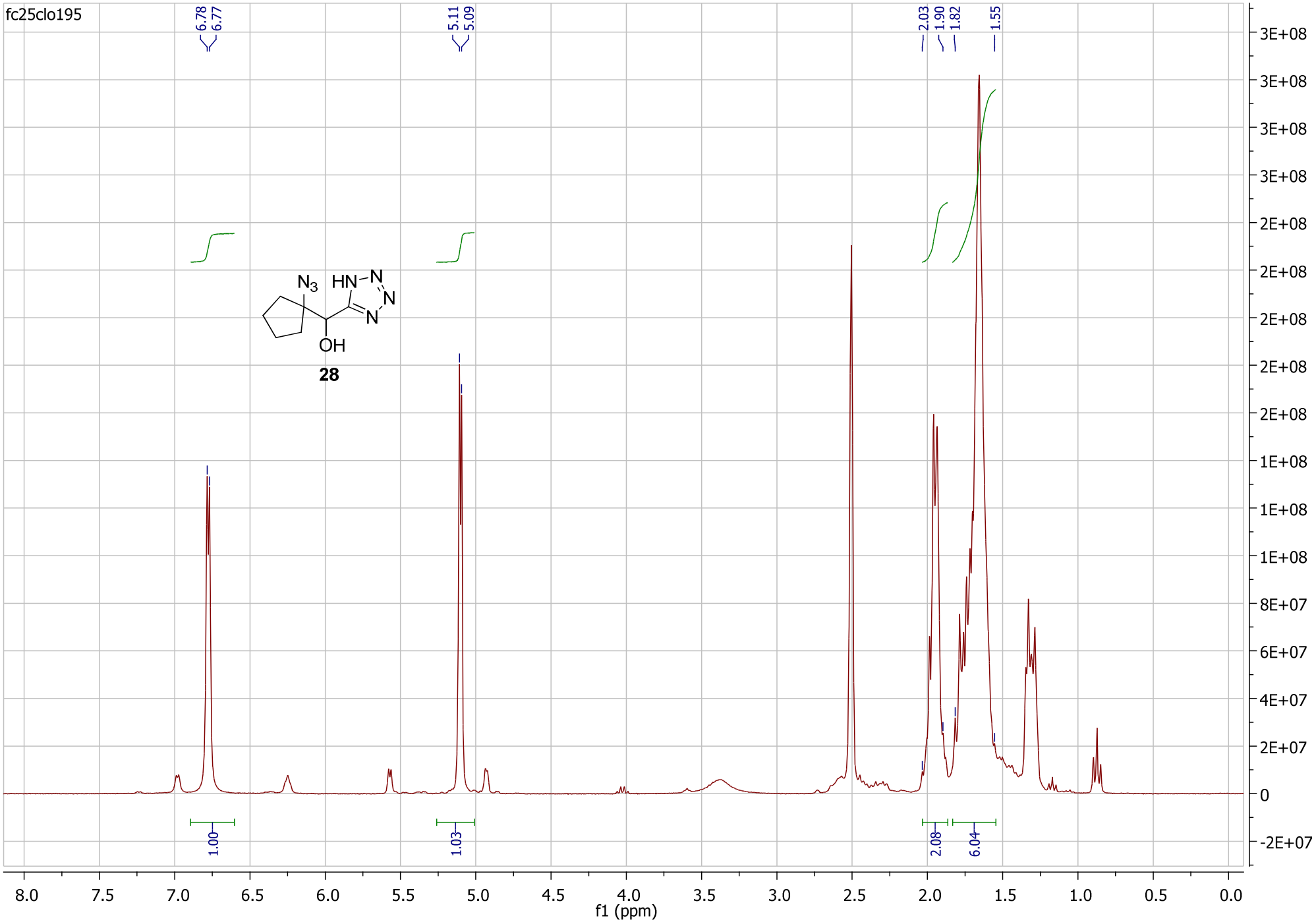

fc16clo177-2
156.33
135.52
135.07
134.89
128.69
128.29
126.63
126.58
122.79
122.27
67.34
67.08
66.68
66.51
N3
HN
N
N
N
OH
25
76
9E+07
8E+07
7E+07
6E+07
5E+07
4E+07
3E+07
2E+07
1E+07
0
-1E+07
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

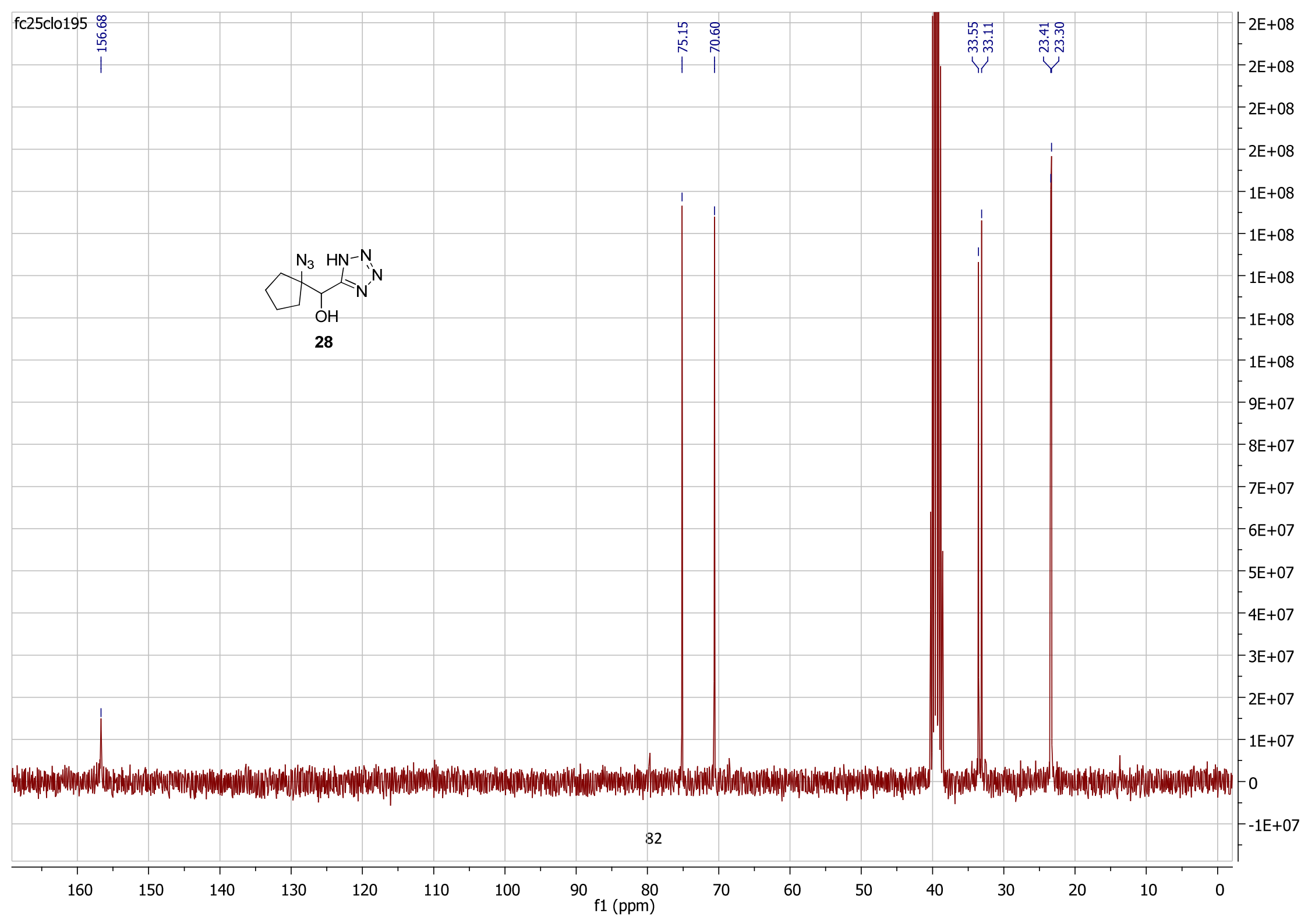

fc20clo183
6.69
5.15
5.14
3.81
3.79
3.78
1.52
1.11
0.95
0.71
N3
HN
N
N
N
OH
26
0.89
1.05
1.00
12.86
3.71
3E+08
2E+08
1E+08
8E+07
6E+07
4E+07
2E+07
0
-2E+07
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

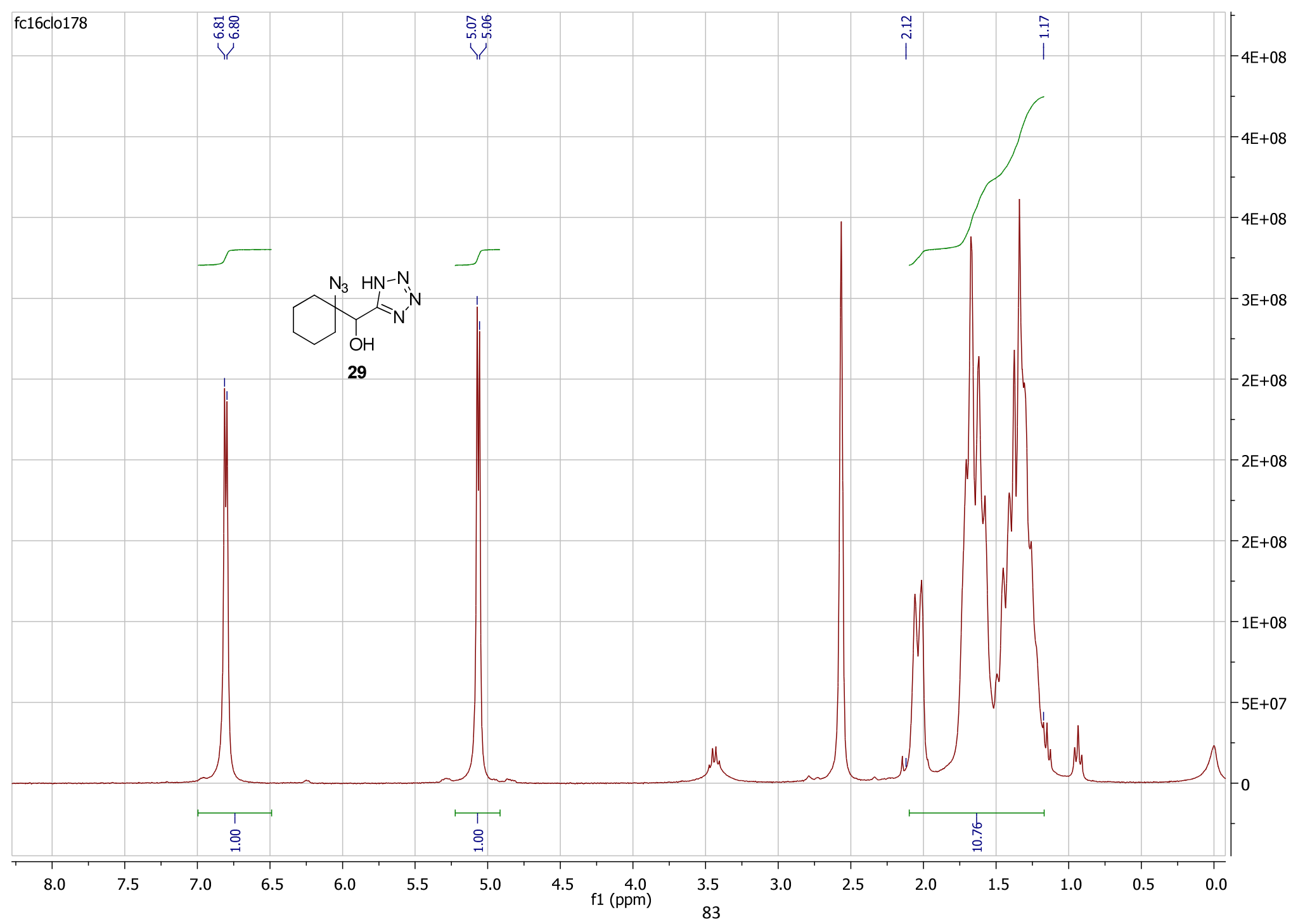

fc20ch183
156.12
67.31
65.57
31.10
29.01
28.56
28.45
25.51
22.02
13.88
N3
HN
N
N
N
OH
26
78
2E+08
2E+08
2E+08
2E+08
2E+08
1E+08
1E+08
1E+08
8E+07
6E+07
4E+07
2E+07
0
-2E+07
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

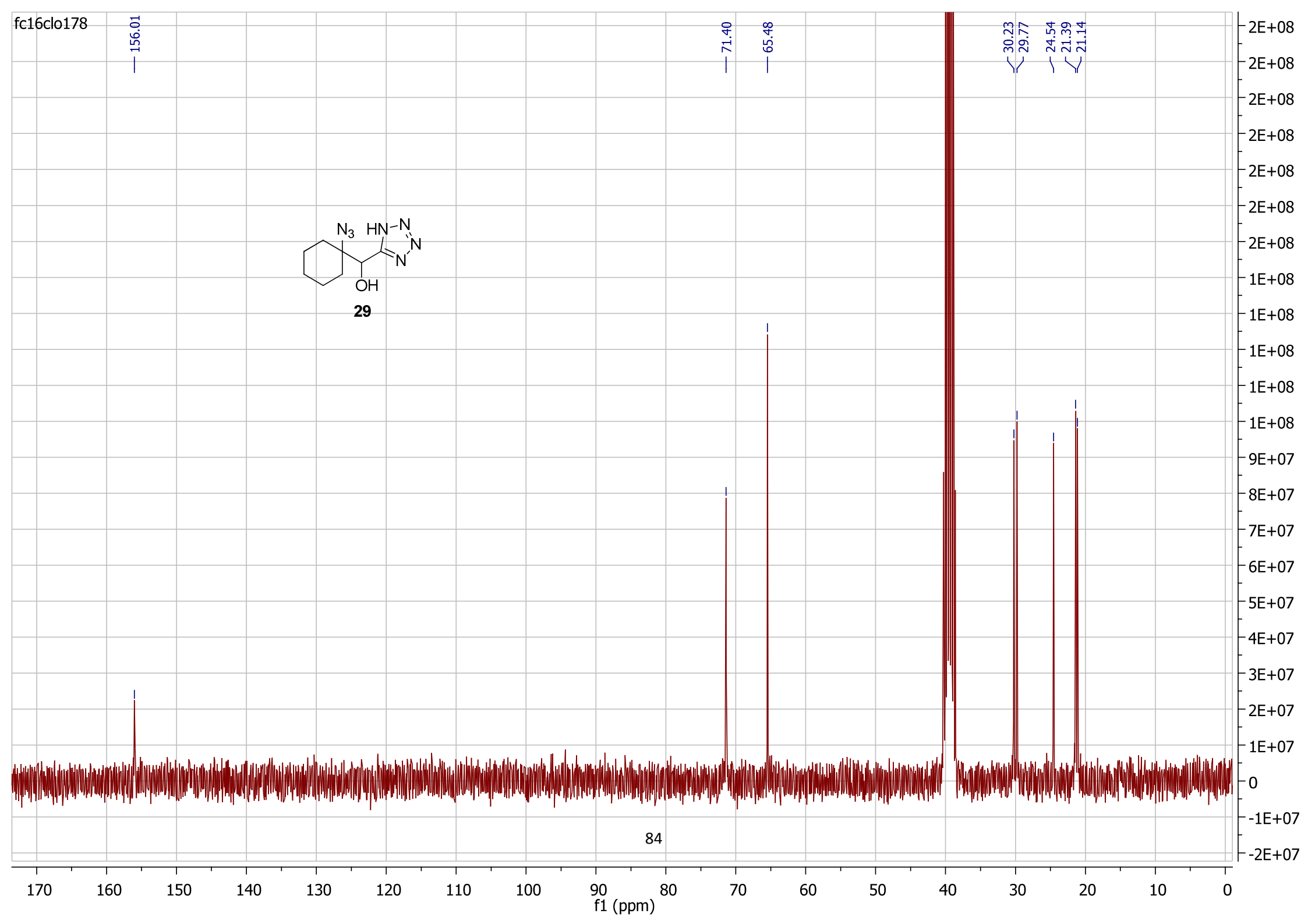

fc28clo203
6.75
6.73
5.10
5.09
1.96
1.94
1.91
1.89
1.87
1.84
1.81
1.79
1.77
1.74
1.72
1.69
1.35
1.33
1.31
1.28
1.25
1.22
1.20
1.17
1.15
0.97
0.94
0.92
0.83
0.80
0.78
N3
HN
N
N
N
OH
27
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
0.96
1.00
2.06
2.26
3.01
3.07
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

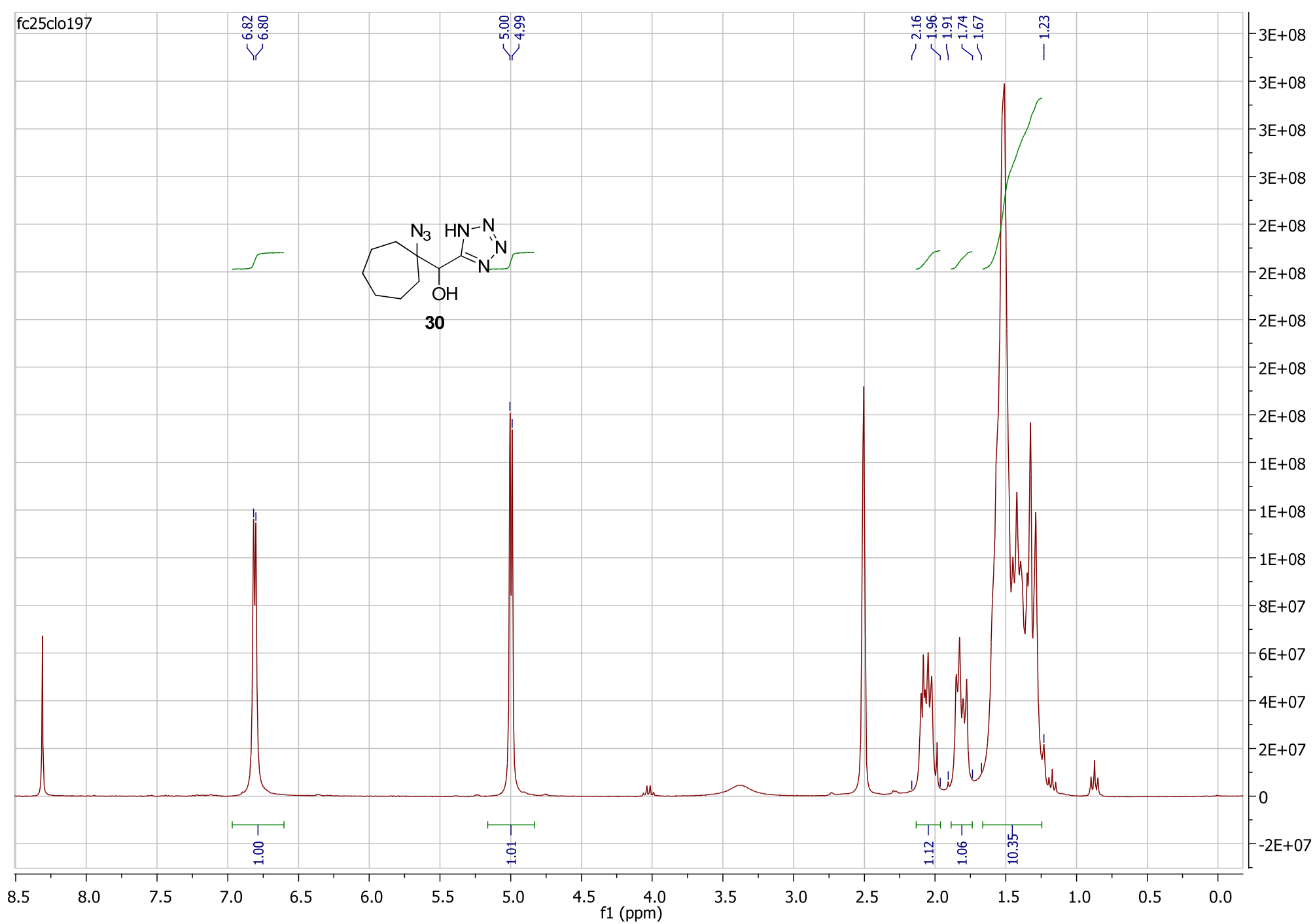

fc28clo203
156.27
68.60
24.24
23.04
7.55
7.36
N3
HN
N
N
N
OH
27
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
-5E+07
80
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

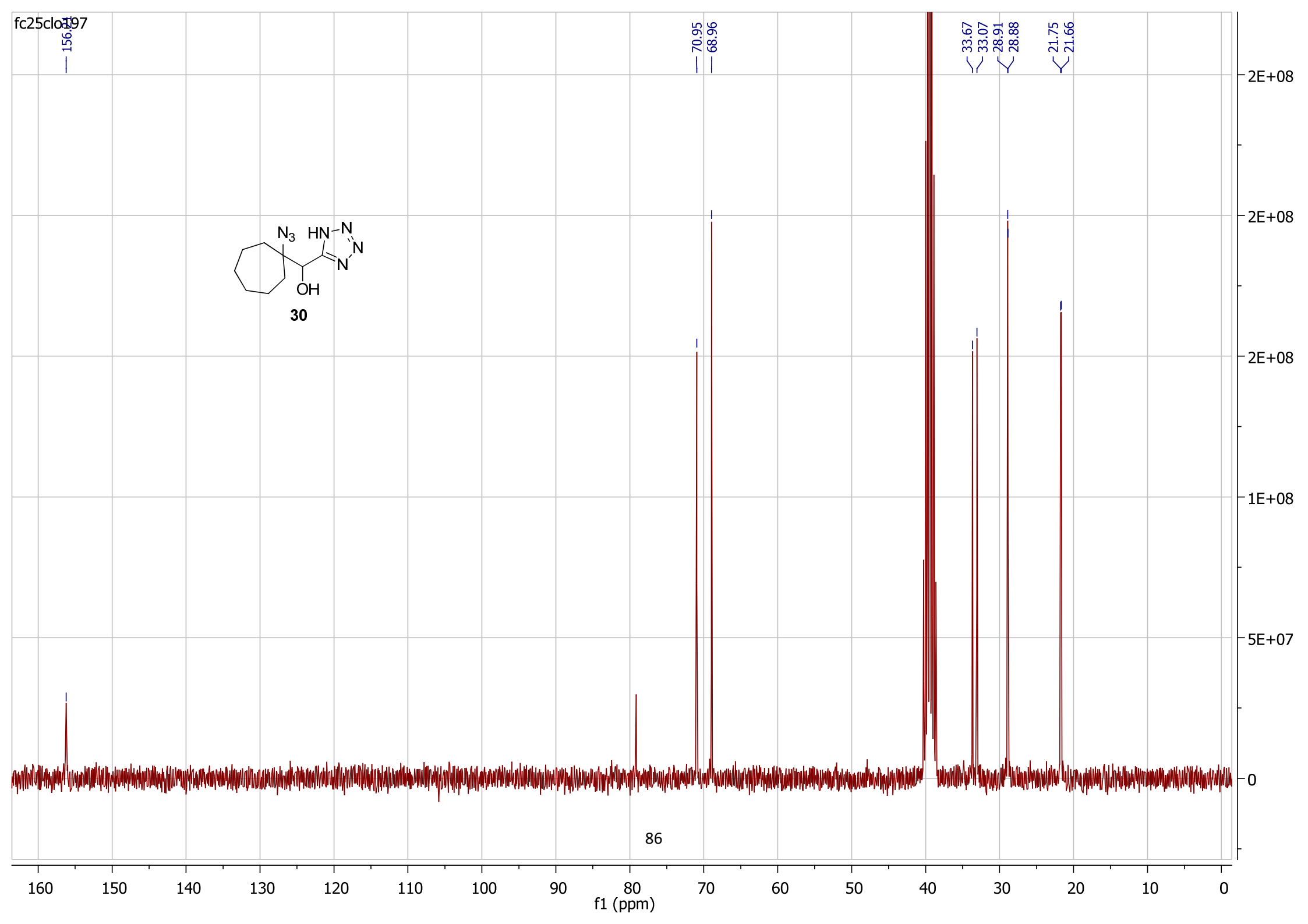

fc25clo195
6.78
6.77
5.11
5.09
2.03
1.90
1.82
1.55
N3
HN
N
N
N
OH
28
1.00
1.03
2.08
6.04
3E+08
2E+08
1E+08
8E+07
6E+07
4E+07
2E+07
0
-2E+07
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

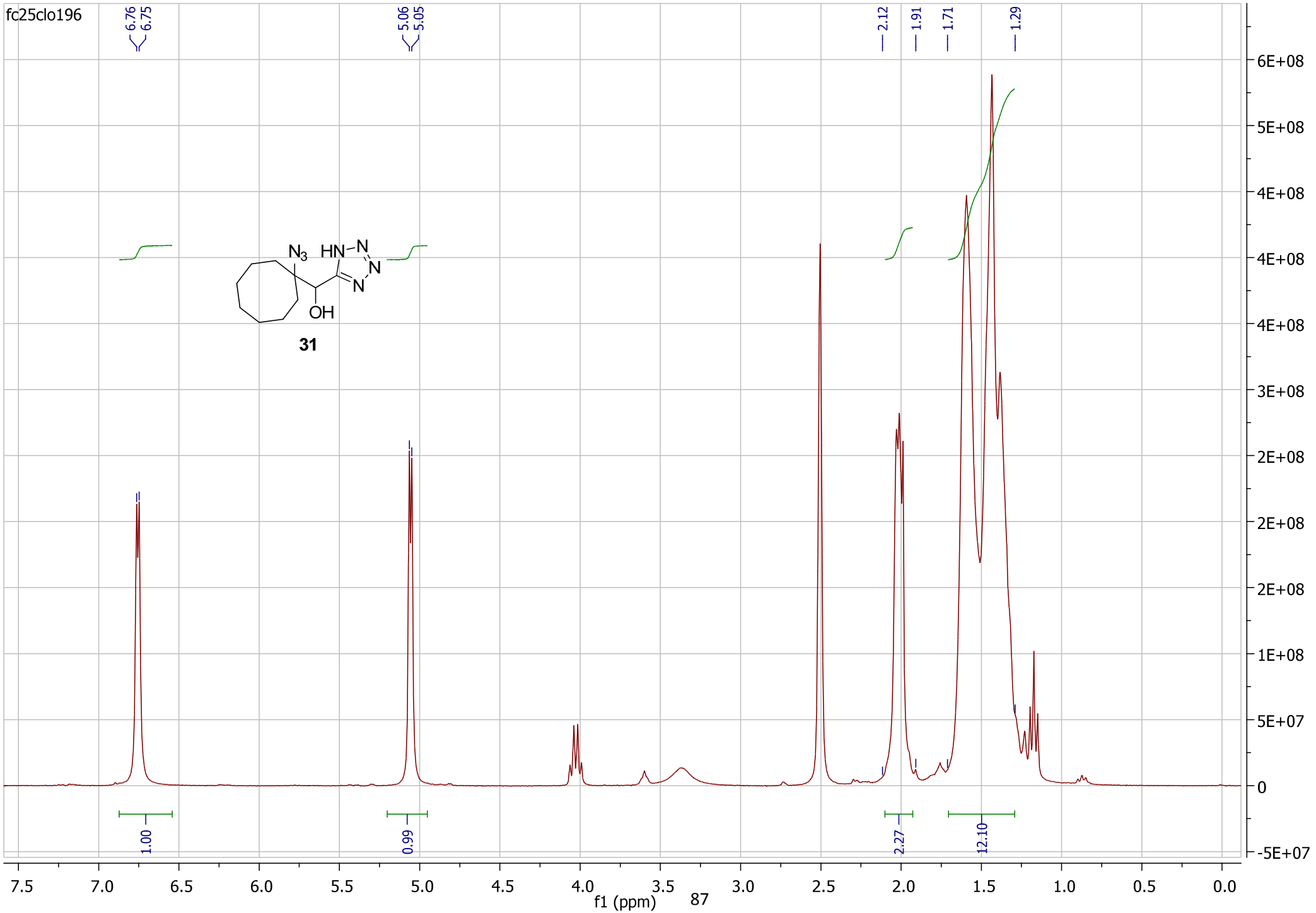

fc25clo195
156.68
75.15
70.60
33.55
33.11
23.41
23.30
N3
HN
N
N
N
OH
28
82
f1 (ppm)

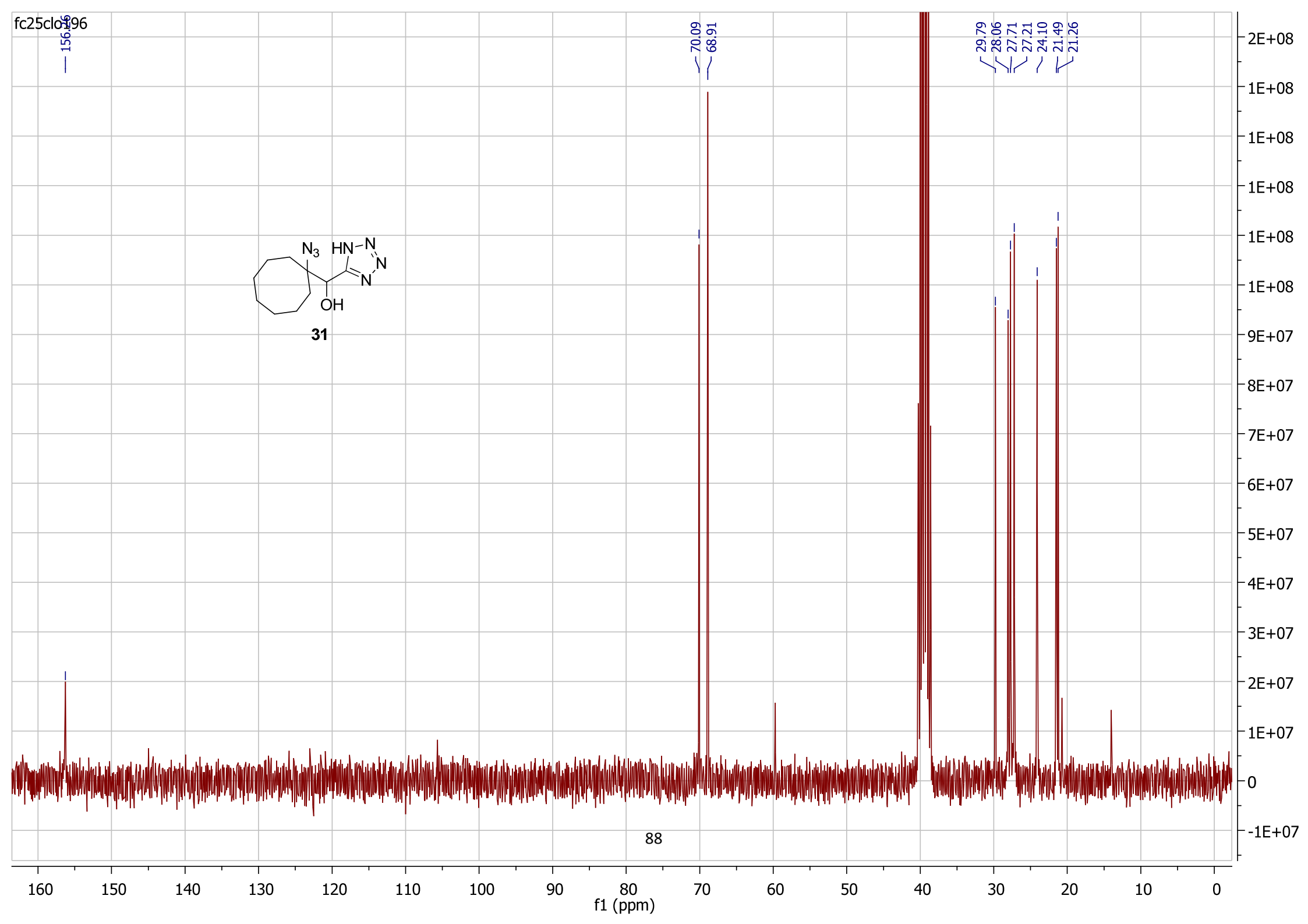

fc16clo178
6.81
6.80
5.07
5.06
2.12
1.17
N3
HN
N
N
N
OH
29
1.00
1.00
10.76
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

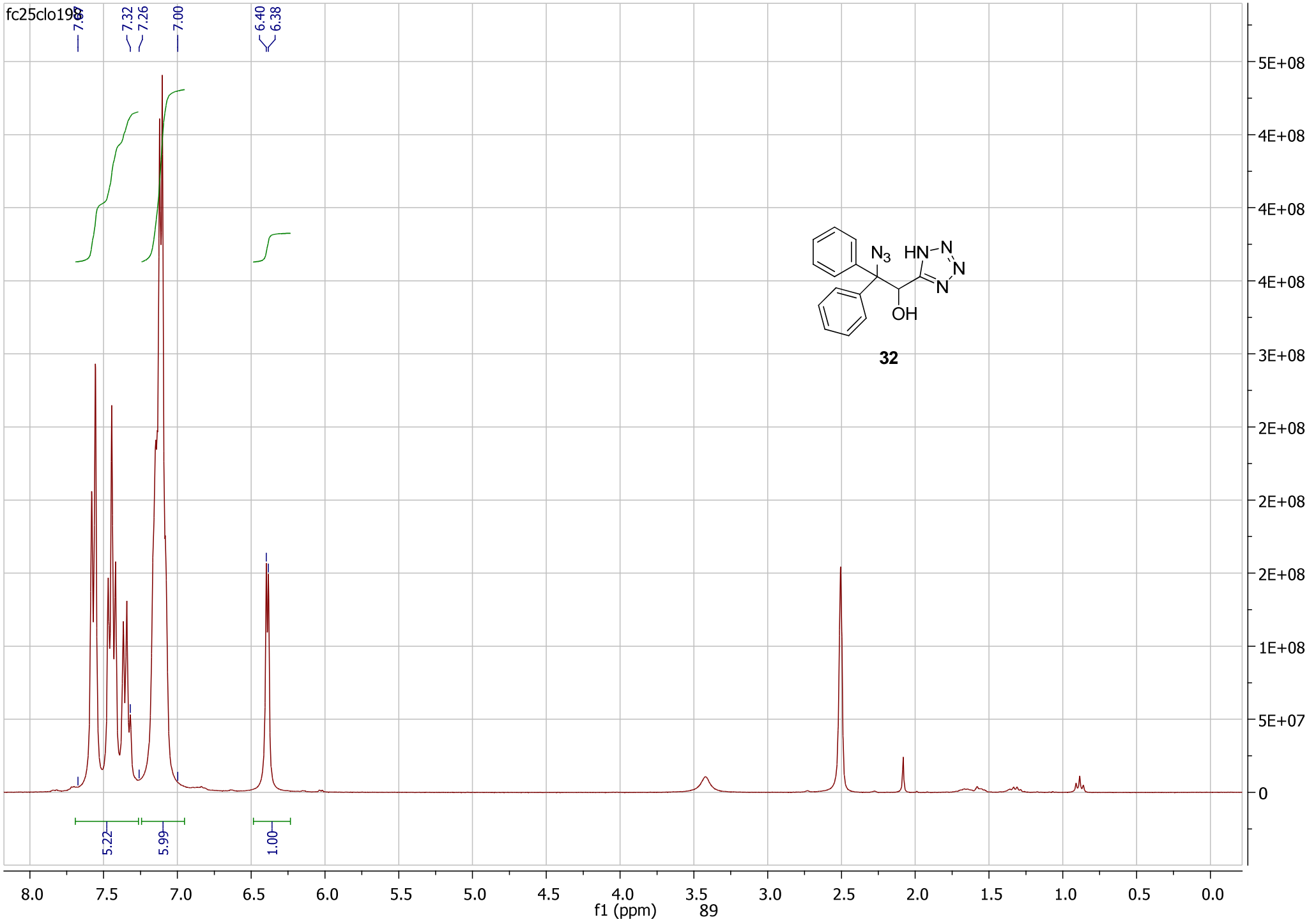

fc16clo178
156.01
71.40
65.48
30.23
29.77
24.54
21.39
21.14
N3
HN
N
N
N
OH
29
84
f1 (ppm)

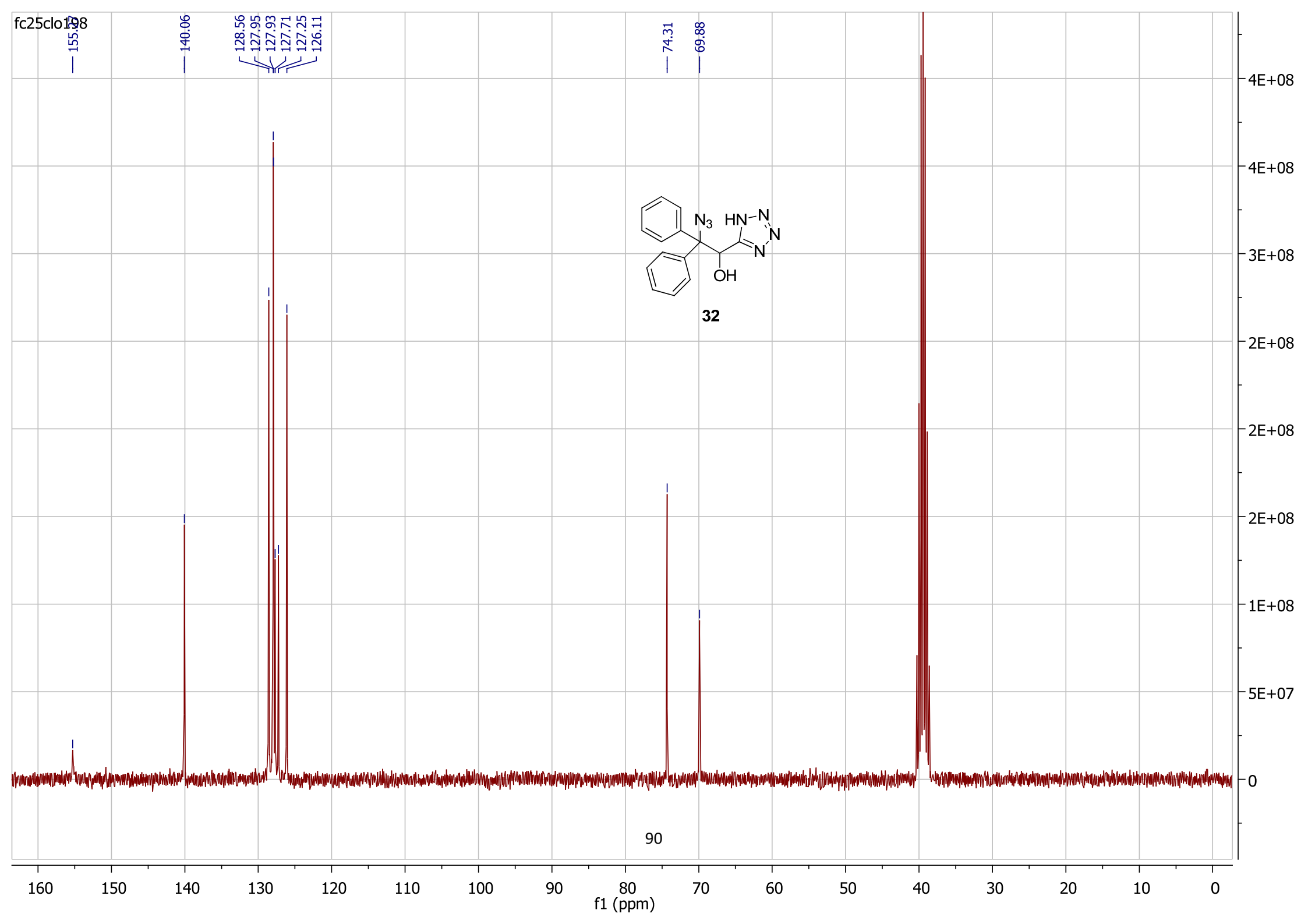

fc25clo197
6.82
6.80
5.00
4.99
2.16
1.96
1.91
1.74
1.67
1.23
N3
HN
N
N
N
OH
30
1.00
1.01
1.12
1.06
10.35
3E+08
2E+08
1E+08
8E+07
6E+07
4E+07
2E+07
0
-2E+07
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

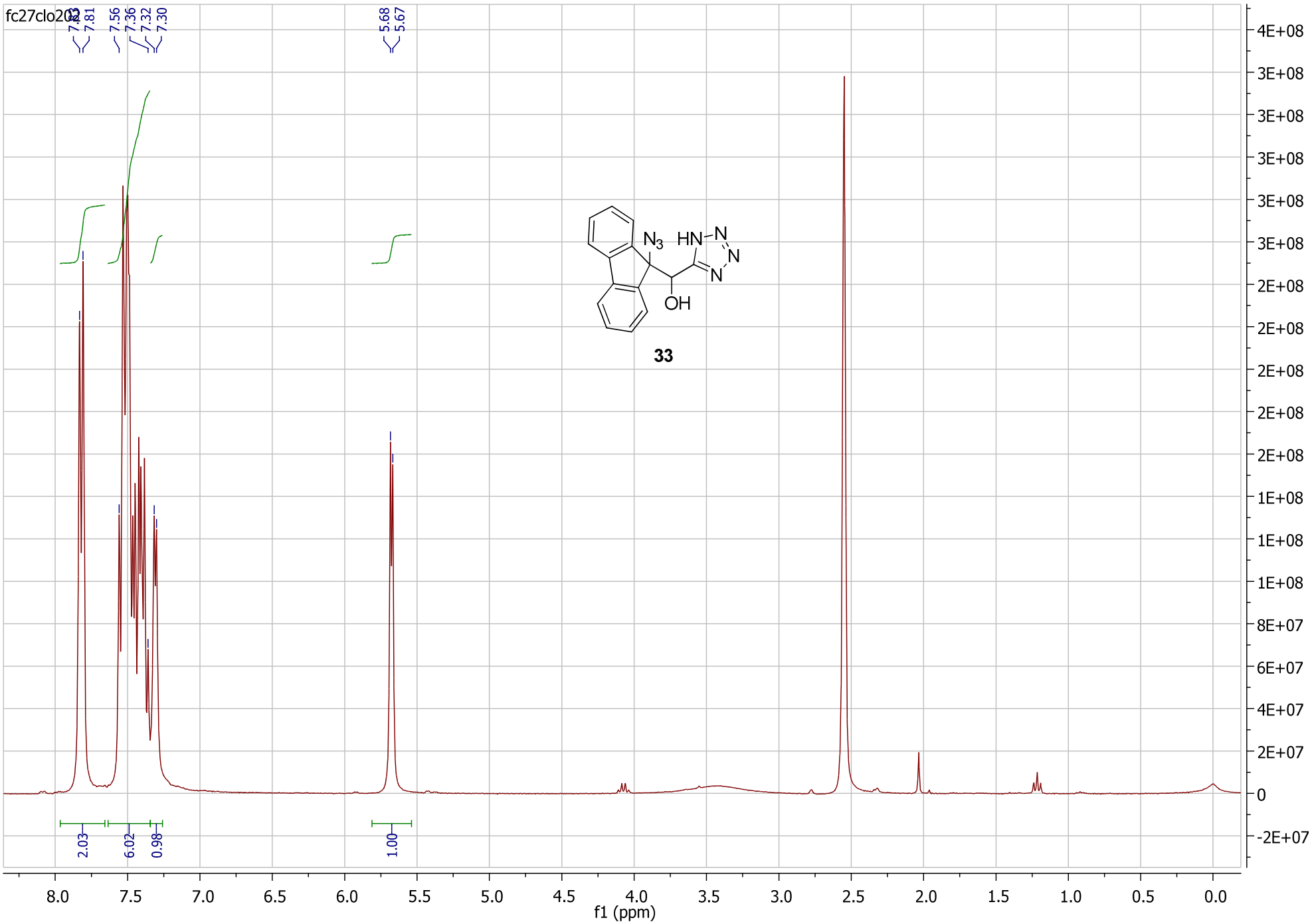

fc25clo197
156.21
70.95
68.96
33.67
33.07
28.91
28.88
21.75
21.66
N3
HN
N
N
N
OH
30
86
2E+08
2E+08
2E+08
1E+08
5E+07
0
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

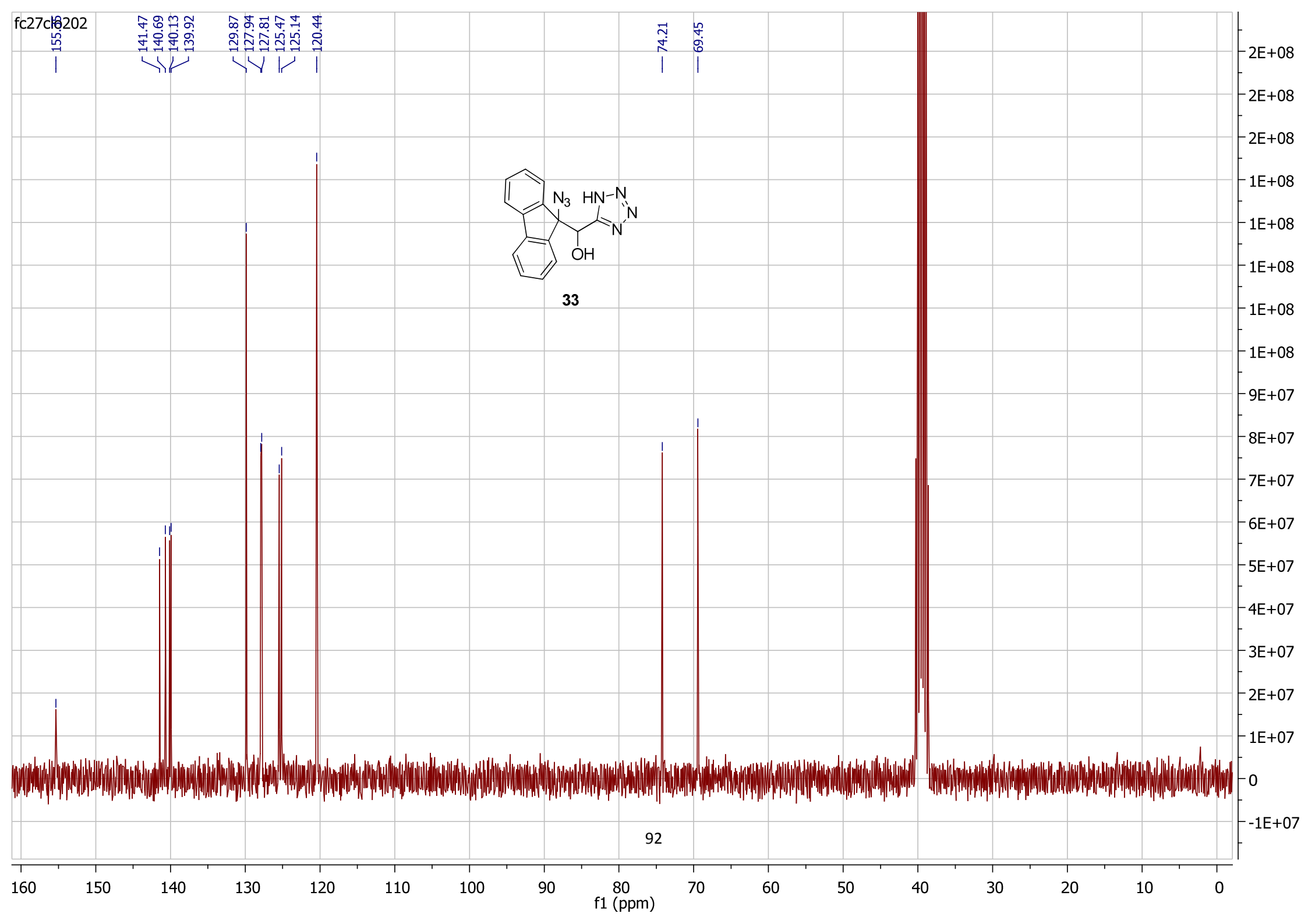

fc25clo196
6.76
6.75
5.06
5.05
2.12
1.91
1.71
1.29
N3
HN
N
N
N
OH
31
1.00
0.99
2.27
12.10
6E+08
5E+08
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
-5E+07
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

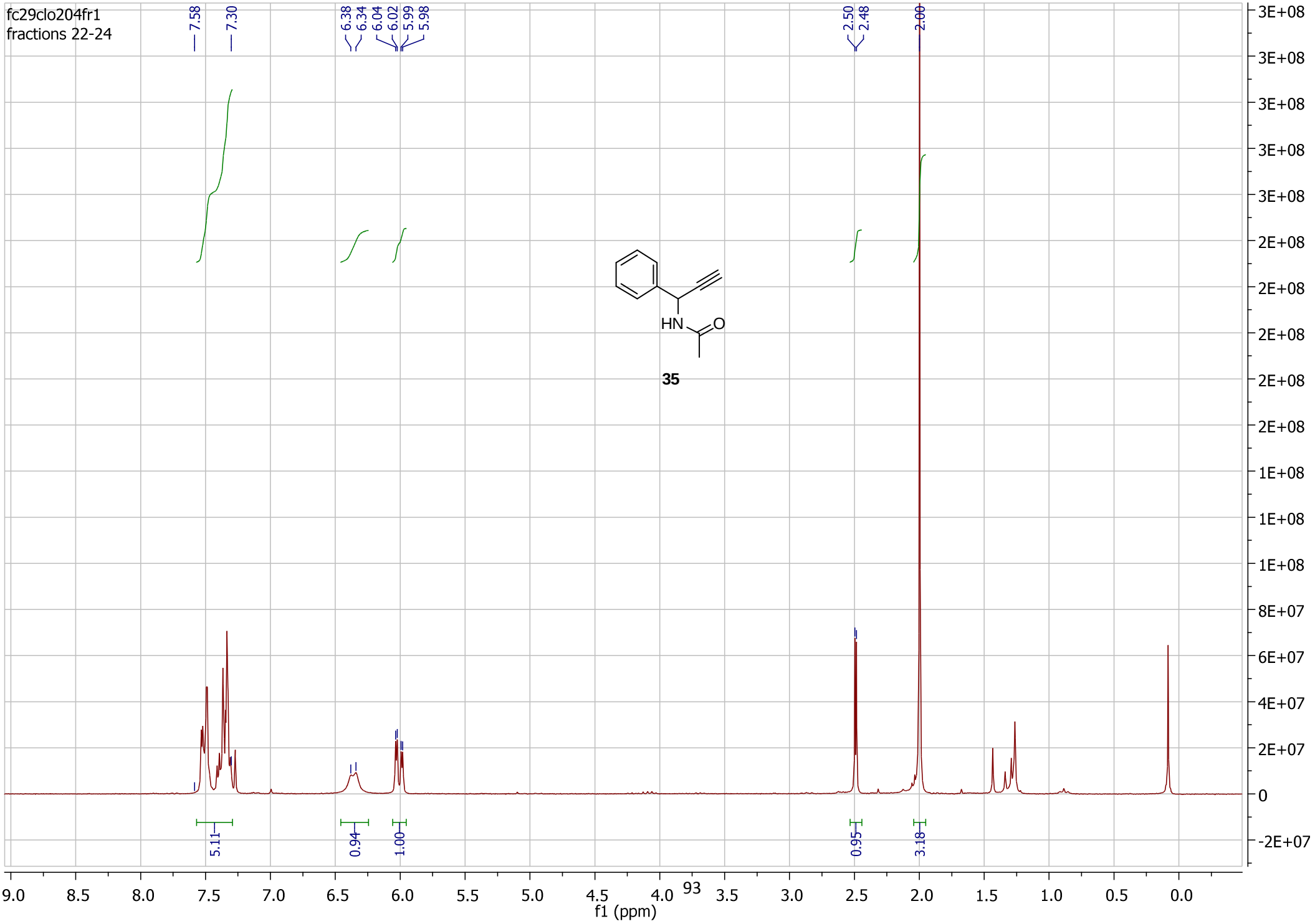

fc25clo196
156.46
70.09
68.91
29.79
28.06
27.71
27.21
24.10
21.49
21.26
N3
HN
N
N
N
OH
31
88
f1 (ppm)

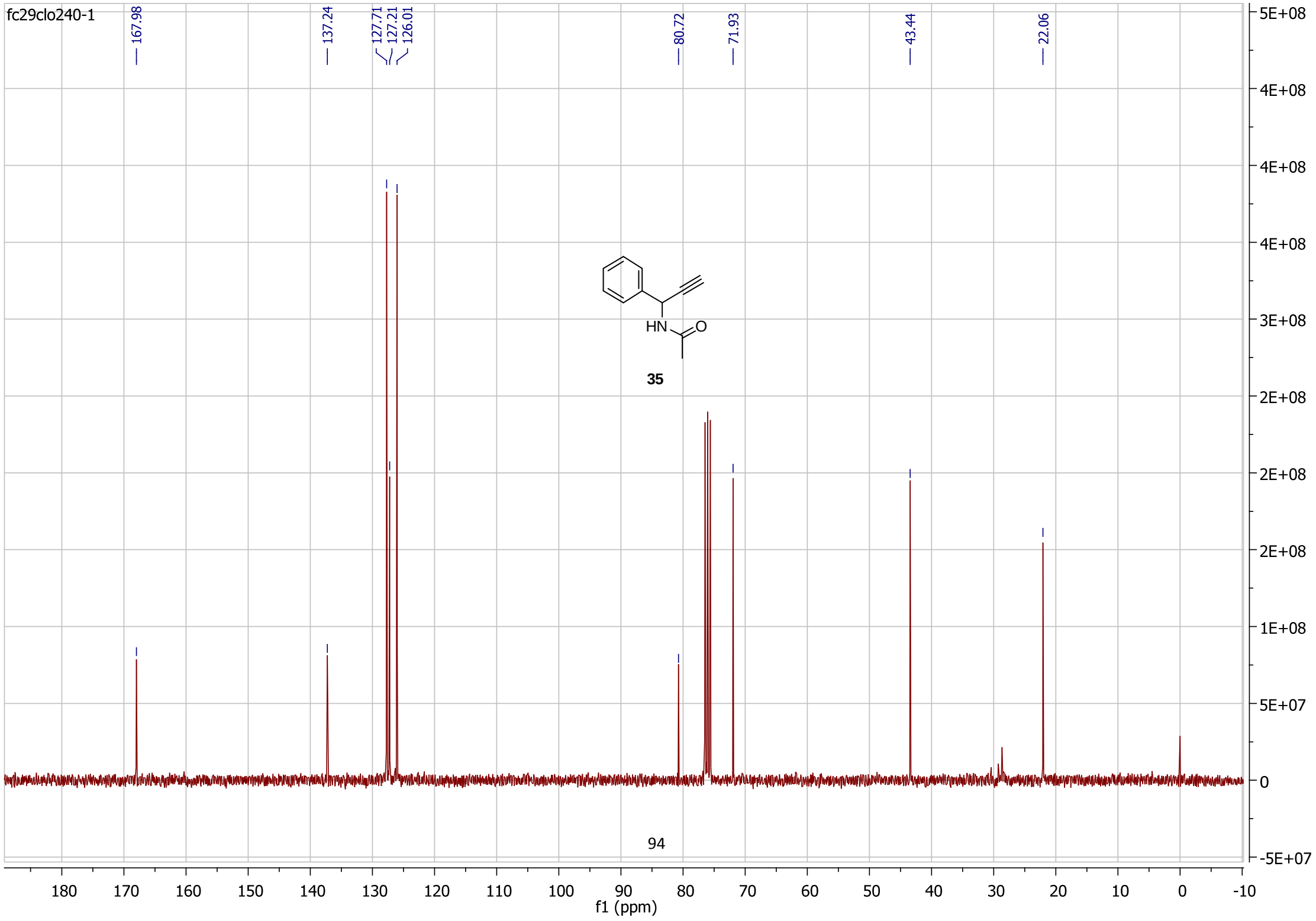

fc25clo1987
7.67
7.32
7.26
7.00
6.40
6.38
5.22
5.99
1.00
N3
HN
N
N
OH
32
5E+08
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

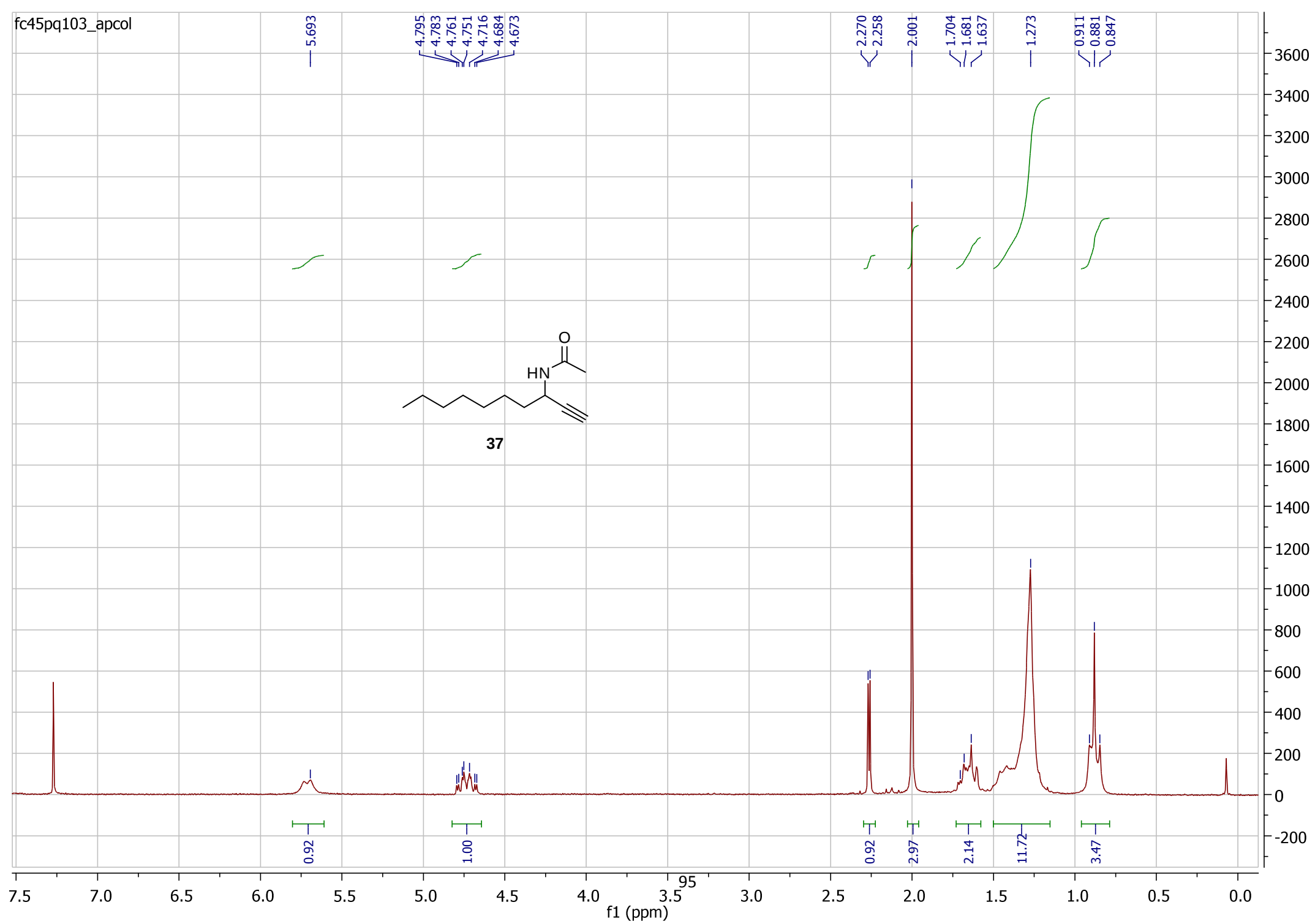

fc25clo1-98
155.19
140.06
128.56
127.95
127.93
127.71
127.25
126.11
74.31
69.88
N3
HN
N
N
N
OH
32
90
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

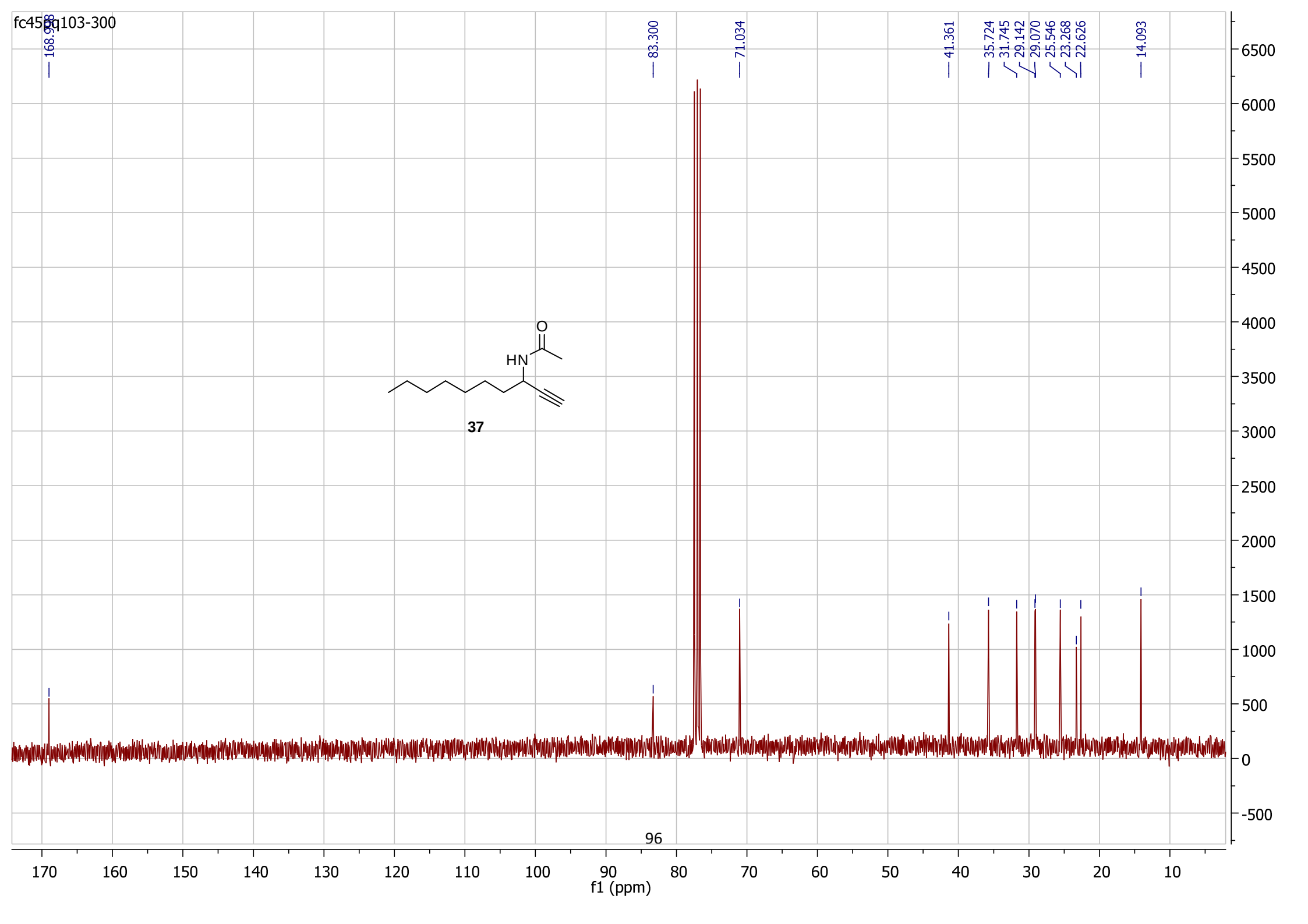

fc27clo202
7.83
7.81
7.56
7.36
7.32
7.30
5.68
5.67
N3
HN
N
N
N
OH
33
2.03
6.02
0.98
1.00
4E+08
3E+08
3E+08
3E+08
3E+08
3E+08
2E+08
2E+08
2E+08
2E+08
2E+08
2E+08
1E+08
1E+08
1E+08
8E+07
6E+07
4E+07
2E+07
0
-2E+07
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

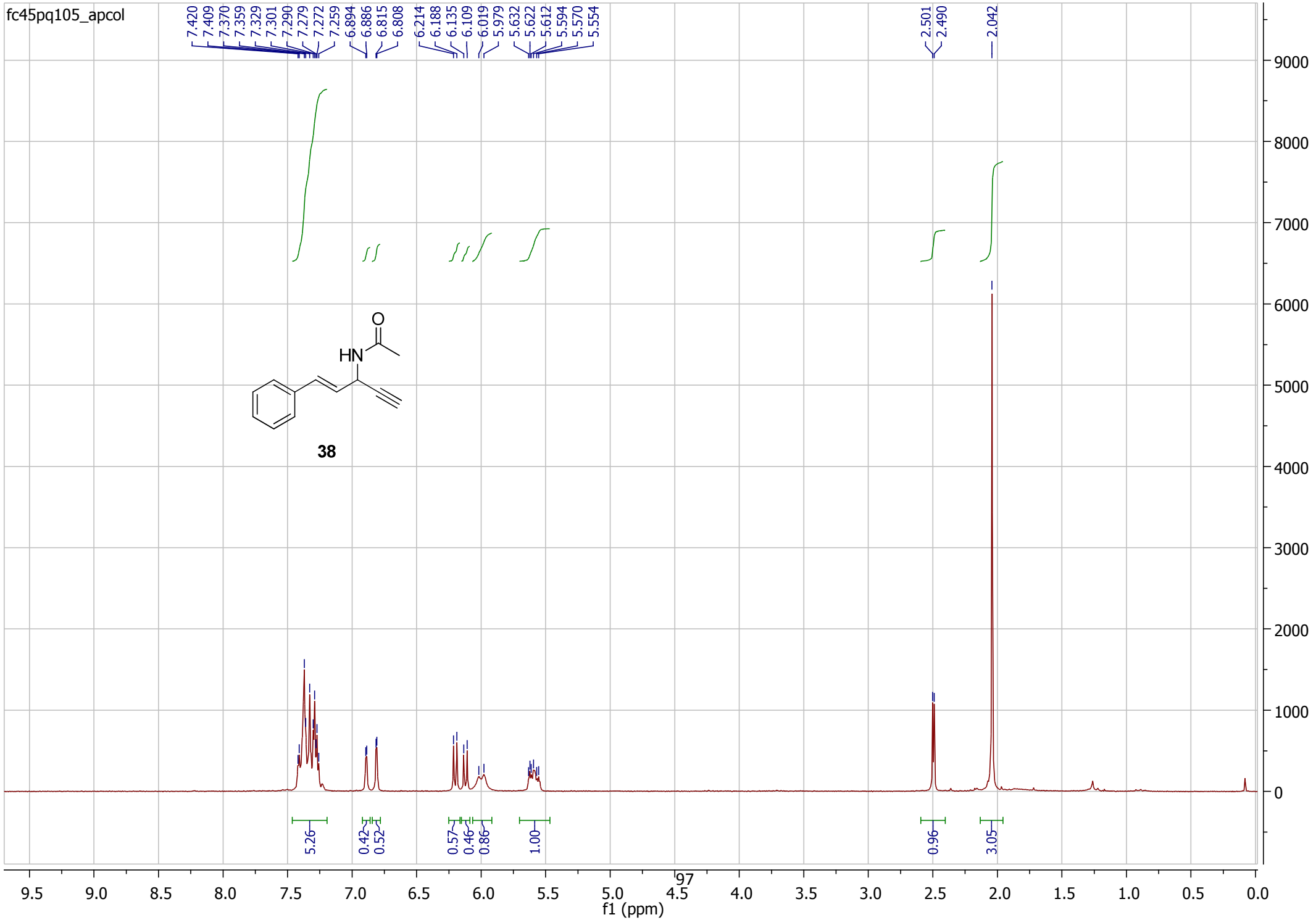

fc27ch6202
155.68
141.47
140.69
140.13
139.92
129.87
127.94
127.81
125.47
125.14
120.44
74.21
69.45
N3
HN
N
N
N
OH
33
92
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)
2E+08
2E+08
2E+08
1E+08
1E+08
1E+08
1E+08
1E+08
1E+08
9E+07
8E+07
7E+07
6E+07
5E+07
4E+07
3E+07
2E+07
1E+07
0
-1E+07

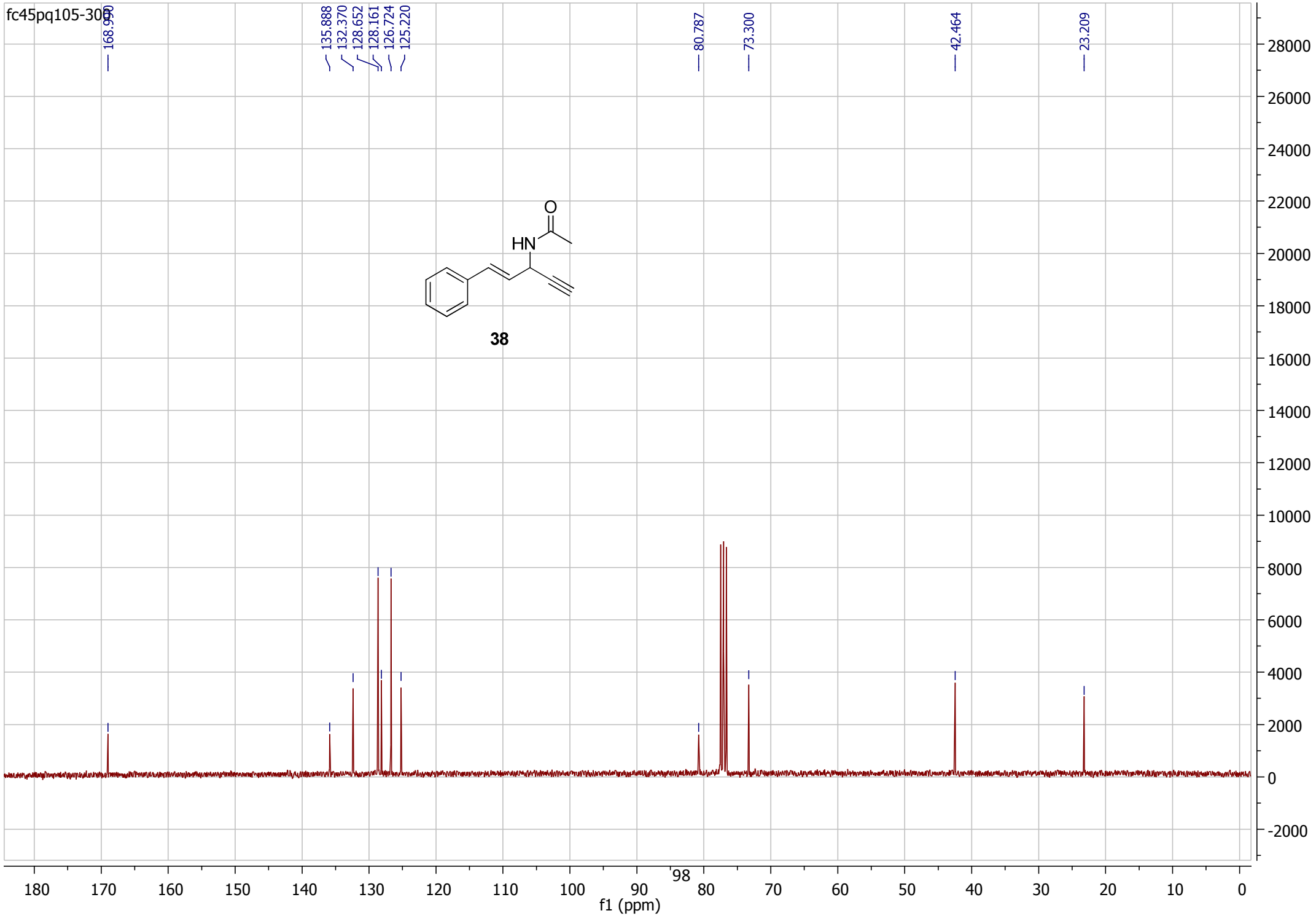

fc29clo204fr1
fractions 22-24
7.58
7.30
6.38
6.34
6.04
6.02
5.99
5.98
2.50
2.48
2.00
HN
O
35
5.11
0.94
1.00
0.95
3.18
3E+08
2E+08
1E+08
8E+07
6E+07
4E+07
2E+07
0
-2E+07
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

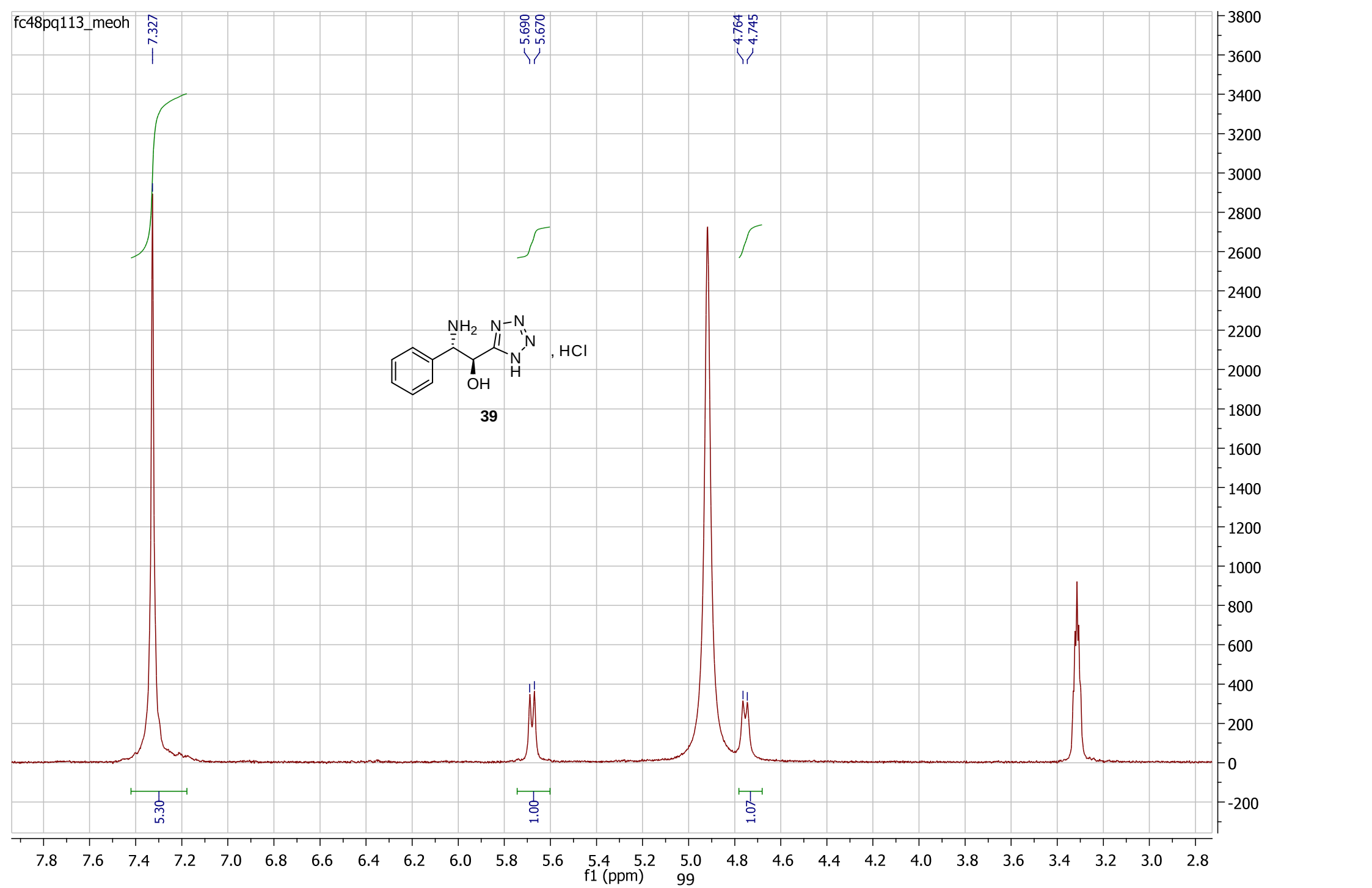

fc29clo240-1
167.98
137.24
127.71
127.21
126.01
80.72
71.93
43.44
22.06
HN
O
35
94
5E+08
4E+08
4E+08
4E+08
3E+08
2E+08
2E+08
2E+08
1E+08
5E+07
0
-5E+07
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

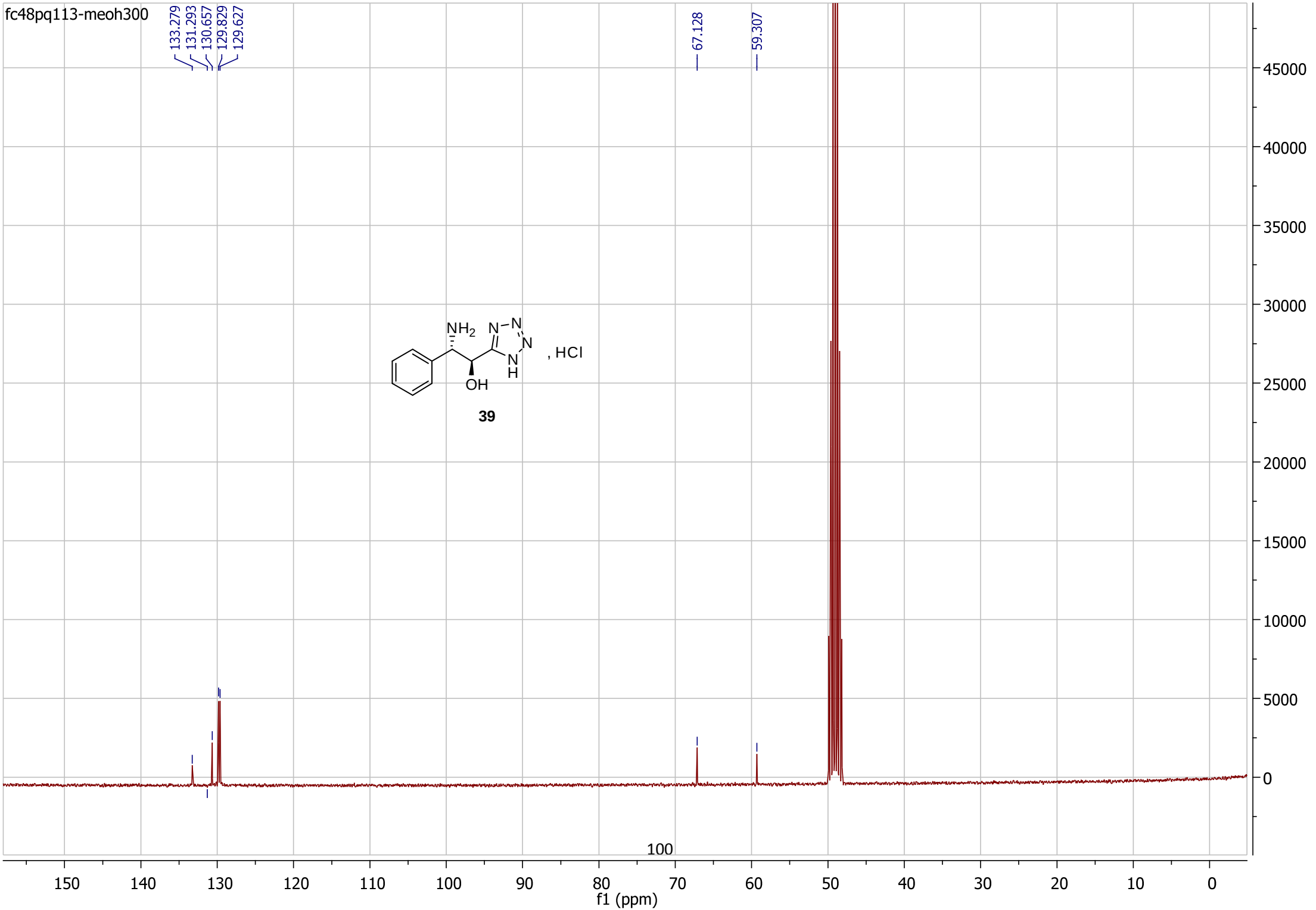

fc45pq103_apcol
5.693
4.795
4.783
4.761
4.751
4.716
4.684
4.673
2.270
2.258
2.001
1.704
1.681
1.637
1.273
0.911
0.881
0.847
O
HN
37
0.92
1.00
0.92
2.97
2.14
11.72
3.47
f1 (ppm)


fc45sq103-300
168.888
83.300
71.034
41.361
35.724
31.745
29.142
29.070
25.546
23.268
22.626
14.093
O
HN
37
96
f1 (ppm)


fc45pq105_apcol
7.420
7.409
7.370
7.359
7.329
7.301
7.290
7.279
7.272
7.259
6.894
6.886
6.815
6.808
6.214
6.188
6.135
6.109
6.019
5.979
5.632
5.622
5.612
5.594
5.570
5.554
2.501
2.490
2.042
HN
O
38
5.26
0.42
0.52
0.57
0.46
0.86
1.00
0.96
3.05
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


fc45pq105-300
168.990
135.888
132.370
128.652
128.161
126.724
125.220
80.787
73.300
42.464
23.209
O
HN
38
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


fc48pq113_meoh
7.327
5.690
5.670
4.764
4.745
NH2
N
N
N
H
OH
, HCl
39
5.30
1.00
1.07
f1 (ppm)


fc48pq113-meoh300
133.279
131.293
130.657
129.829
129.627
67.128
59.307
NH2
N
N
N
H
OH
, HCl
39
100
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)