Electronic Supplementary Information

Fe-catalyzed three-component carboazidation of alkenes with alkanes and trimethylsilyl azide

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I. General information

Unless otherwise noted, all commercially available reagents and analytical grade solvents (cyclohexane, cyclopentane, cyclooctane, cycloheptane, hexane, pentane, dichloromethane, chloroform, tetrachloromethane) were used as received without further purification.

Thin-layer chromatography (TLC) was performed using E. Merck silica gel 60 F254 precoated plates (0.25 mm) or Sorbent Silica Gel 60 F254 plates and visualized by UV lamp (254 nm). High-resolution mass spectra (HRMS) were obtained from a JEOL JMS-700 instrument (ESI) or Thermo Scientific LTQ-Orbitrap XL (MALDI). Melting points are uncorrected. Nuclear magnetic resonance (NMR) spectra were recorded on a Bruker Avance 400 spectrometer at ambient temperature. Chemical shifts for ^{1}H NMR spectra are reported in parts per million (ppm) from tetramethylsilane with the solvent resonance as the internal standard (chloroform: δ 7.26 ppm). Chemical shifts for ^{13}C NMR spectra are reported in parts per million (ppm) from tetramethylsilane with the solvent as the internal standard ($CDCl_3$: δ 77.16 ppm). Data are reported as following: chemical shift, multiplicity (s = singlet, d = doublet, dd = doublet of doublets, t = triplet, q = quartet, m = multiplet, br = broad signal), coupling constant (Hz), and integration.

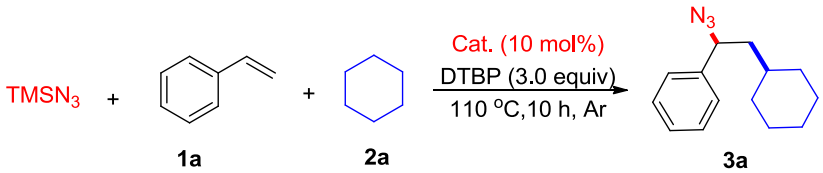
II. General experimental procedure

Representative procedure for the Fe-catalyzed three-component carboazidation of alkene:

An oven-dried reaction vessel was charged with $Fe(acac)_3$ (0.001 mmol, 0.5 mol%) in cyclohexane (1.5 mL, **2a**, pre-prepared solution), styrene (**1a**, 0.2 mmol, 1.0 equiv), $TMSN_3$ (0.5 mmol, 2.5 equiv) and DTBP (0.5 mmol, 2.5 equiv) under argon atmosphere. The vessel was sealed and the mixture was stirred with heating at 110 °C (oil bath temperature) for 10 h. Afterwards the resulting mixture was cooled to room temperature. The solvent was removed in vacuo and the residue was purified by column chromatography on silica gel using a mixture of dichloromethane/petroleum ether as eluent to give product **3a**.

III. Conditions optimization

Table S1. Optimization of catalysts[a]

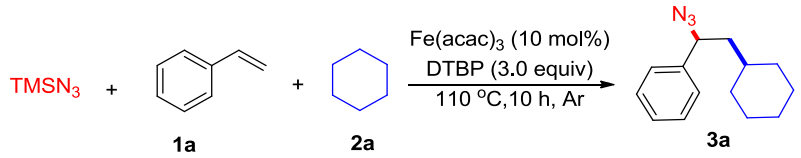


entry	Cat. (mol%)	yield [%][b]
1	-	7
2	$FeCl_2$	<2
3	$FeCl_3$	29
4	$Fe(acac)_3$	53

5	**$Fe(acac)_2$**	**42**
6	**$Fe(OAc)_2 \cdot 4H_2O$**	**n.d.**
7	**$Co(acac)_3$**	**n.d.**
8	**$Mn(AcO)_2$**	**n.d.**
9	**$Cu(acac)_2$**	**n.d.**
10	**$Cu(OTf)_2$**	**n.d.**
11	**CuCl**	**n.d.**

[a] Reaction conditions: styrene (**1a**, 0.2 mmol, 1.0 equiv), cyclohexane (**2a**, 1.0 mL), $TMSN_3$ (0.5 mmol, 2.5 equiv), DTBP (0.6 mmol, 3.0 equiv), Cat. (0.02 mmol, 10 mol %), stirred at 110 ℃ for 10 h under Ar. [b] Isolated yields.

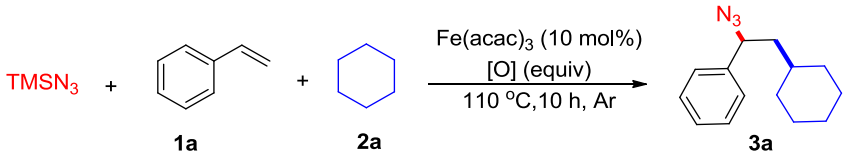
Table S2. Optimization for the amount of cyclohexane [a]



entry	cyclohexane (X mL)	yield [%] [b]
1	**1.0**	**53**
2	**1.5**	**68**
3	**2.0**	**62**

[a] Reaction conditions: styrene (**1a**, 0.2 mmol, 1.0 equiv), cyclohexane (**2a**, X mL), $TMSN_3$ (0.5 mmol, 2.5 equiv), DTBP (0.6 mmol, 3.0 equiv), $Fe(acac)_3$ (0.02 mmol, 10 mol %), stirred at 110 ℃ for 10 h under Ar. [b] Isolated yields.

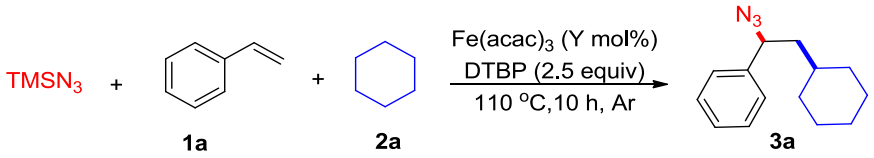
Table S3. Optimization of oxidants [a]



Entry	[O] (equiv)	yield [%][b]
1	**DTBP (3.0)**	**68**
2	**TBHP in Decane (3.0)**	**17**
3	**$PhI(AcO)_2$ (3.0)**	**n.d.**
4	**$K_2S_2O_8$ (3.0)**	**n.d.**
5	**DCP (3.0)**	**37**
6	**BPO (3.0)**	**35**
7	**DTBP (2.5)**	**73**
8	**DTBP (2.0)**	**65**

[a] Reaction conditions: styrene (**1a**, 0.2 mmol, 1.0 equiv), cyclohexane (**2a**, 1.5 mL), $TMSN_3$ (0.5 mmol, 2.5 equiv), [O] (equiv), $Fe(acac)_3$ (0.02 mmol, 10 mol %), stirred at 110 ℃ for 10 h under Ar. [b] Isolated yields.

Table S4. Optimization of the amount of catalyst [a]



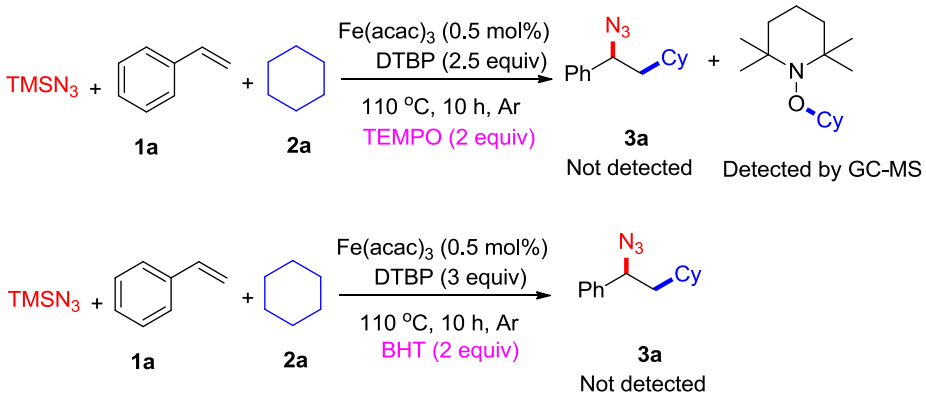
entry	$Fe(acac)_3$ (Y mol%)	yield [%][b]
1	15	64
2	10	73
3	5	72
4	1	72
5	0.5	74
6	0.25	69

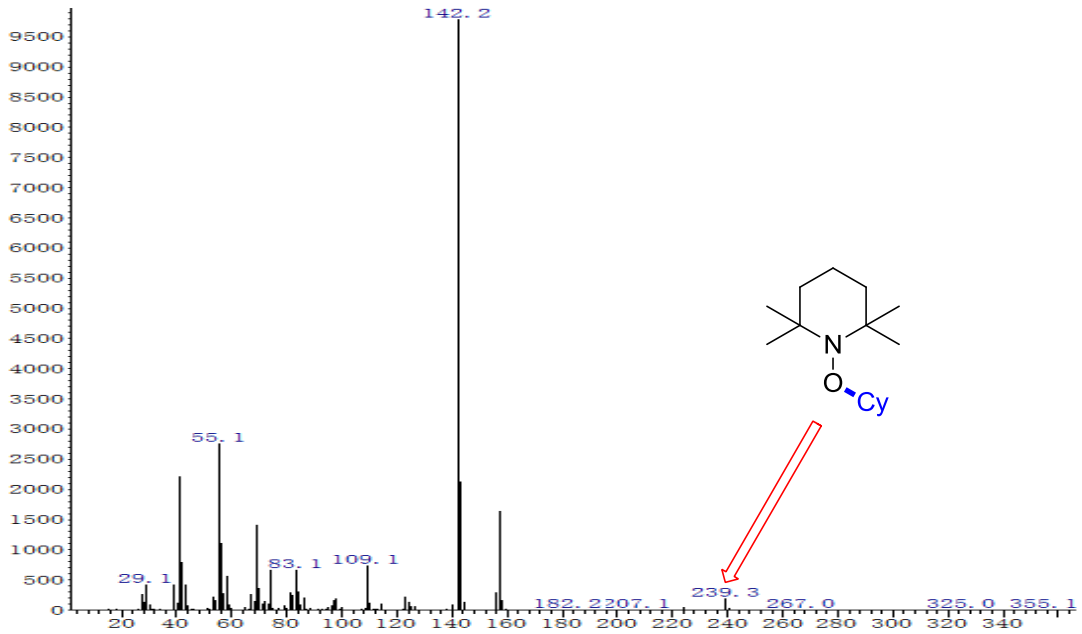
[a] Reaction conditions: styrene (**1a**, 0.2 mmol, 1.0 equiv), cyclohexane (**2a**, 1.5 mL), $TMSN_3$ (0.5 mmol, 2.5 equiv), DTBP (0.5 mmol, 2.5 equiv), $Fe(acac)_3$ (Y mol %), stirred at 110 ℃ for 10 h under Ar. [b] Isolated yields.

IV. Mechanistic study

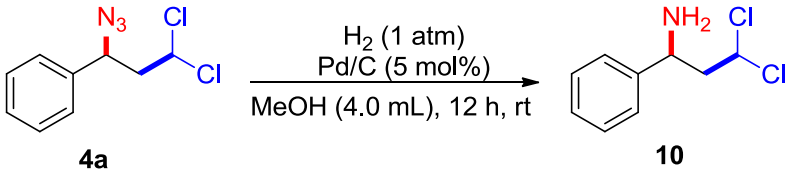
Radical capture experiment:

To further understand this Fe-catalyzed three-component alkylation-azidation of styrenes with alkane and trimethylsilyl azide, several control experiments were carried out in the presence of various radical scavengers. We found that: (a) in the presence of TEMPO (2,2,6,6-tetramethylpiperidine-1-oxyl, 2.0 equiv), the reaction was completely inhibited and the 1-(cyclohexyloxy)-2,2,6,6-tetramethylpiperidine was detected by GC-MS, which supported our speculation that the reaction proceeded via a radical pathway and confirmed the generation of alkyl radical; (b) in the presence of BHT (2,6-di-tert-butyl-4-methylphenol, 2.0 equiv), the product **3a** was not detected, which further confirmed that this cascade reaction was realized via radical pathway.



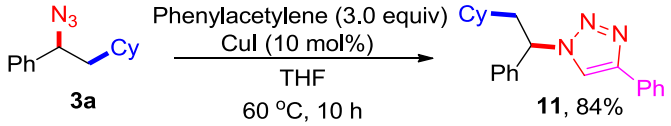


V. Synthetic transformations of the carboazidation products



4a **10**

To a solution of **4a** (91.6 mg, 0.4 mmol) in MeOH (4 mL, 0.1 M) was added Pd/C (m/m 10%, 50% wet, 42 mg, 5 mol%). The reaction mixture was evacuated and filled with H_2 (from balloon) three times. After being stirred at room temperature for 12 hours, the reaction mixture was filtered through a plug of Celite. The resulting filtrate was concentrated in vacuo. The crude product was purified by flash column chromatography on silica gel (CH_2Cl_2/MeOH = 40:1) to give compound **14** (colorless oil, 57.6 mg, 71%).

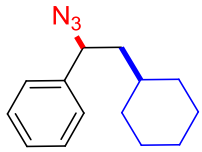


3a **11**, 84%

Compound **3a** (0.2 mmol,1.0 equiv) was dissolved in THF (l mL) in an oven-dried glass tube. Then phenylacetylene (0.6 mmol, 3.0 equiv) and CuI (0.02 mmol, 0.1 equiv) were added. The reaction mixture was stirred at 60 ℃ for 10 h, and the resulting solution was concentrated under vacuum. The residue was purified by column chromatography on silica gel with an eluent of petroleum ether and ethyl acetate (5:1) to afford compound **15** as a white solid (55.6 mg, 84%).

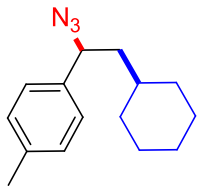
VI. Spectroscopic data of the products

(3a) (1-azido-2-cyclohexylethyl)benzene



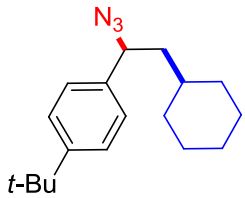
The title compound was prepared according to the general procedure described above by the reaction of styrene (**1a**) with $TMSN_3$ and cyclohexane (**2a**), and purified by flash column chromatography as colorless oil (33.8 mg, 74%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.28 (m, 5H), 4.51 (dd, *J* = 8.4, 6.4 Hz, 1H), 1.80 – 1.53 (m, 7H), 1.39 – 1.31 (m, 1H), 1.26 – 1.12 (m, 3H), 0.99 – 0.87 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.3, 128.9, 128.3, 127.0, 63.9, 43.8, 34.5, 33.6, 33.0, 26.6, 26.3, 26.2. IR (cm^{-1}): 2924, 2850, 2097, 1448, 1240, 816. HRMS: calcd. for $C_{14}H_{20}N^+$ $[M–N_2+H]^+$: 202.1596, Found: 202.1590.

(3b) 1-(1-azido-2-cyclohexylethyl)-4-methylbenzene



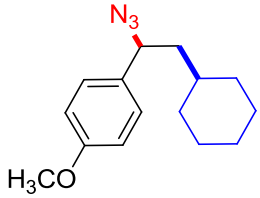
The title compound was prepared according to the general procedure described above by the reaction of 4-methylphenylene (**1b**) with $TMSN_3$ and cyclohexane (**2a**), and purified by flash column chromatography as colorless oil (33.0 mg, 68%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18 (s, 4H), 4.50 – 4.43 (m, 1H), 2.35 (s, 3H), 1.77 – 1.51 (m, 7H), 1.39 – 1.28 (m, 1H), 1.26 – 1.10 (m, 3H), 0.98 – 0.86 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.0, 137.2, 129.6, 127.0, 63.7, 43.7, 34.5, 33.5, 33.0, 26.6, 26.2, 26.1, 21.3. IR (cm^{-1}): 2924, 2851, 2097, 1448, 1241, 816. HRMS: calcd. for $C_{15}H_{22}N^+$ $[M–N_2+H]^+$: 216.1747, Found: 216.1731.

(3c) 1-(1-azido-2-cyclohexylethyl)-4-(*tert*-butyl)benzene



The title compound was prepared according to the general procedure described above by the reaction of 4-*tert*-butylstyrene (**1c**) with $TMSN_3$ and cyclohexane (**2a**), and purified by flash column chromatography as colorless oil (52.4 mg, 92%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.35 (m, 2H), 7.25 – 7.20 (m, 2H), 4.47 (dd, *J* = 9.2, 6.0 Hz, 1H), 1.82 – 1.62 (m, 6H), 1.60 – 1.51 (m, 1H), 1.43 – 1.34 (m, 1H), 1.32 (s, 9H), 1.29 – 1.12 (m, 3H), 1.01 – 0.85 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 151.2, 137.3, 126.6, 125.8, 63.6, 43.8, 34.7, 34.5, 33.6, 32.9, 31.4, 26.6, 26.3, 26.2. IR (cm^{-1}): 2924, 2852, 2097, 1448, 1241, 829. HRMS: calcd. for $C_{18}H_{28}N^+$ $[M–N_2+H]^+$: 258.2222, Found: 258.2156.

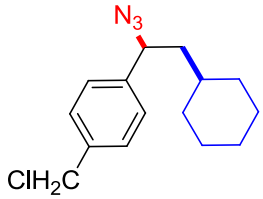
(3d) 1-(1-azido-2-cyclohexylethyl)-4-methoxybenzene



The title compound was prepared according to the general procedure described above by the reaction of 4-methoxystyrene (**1d**) with $TMSN_3$ and cyclohexane (**2a**), and purified by flash column chromatography as colorless oil (28.5 mg, 55%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 – 7.21 (m, 2H), 6.93 – 6.88 (m, 2H), 4.46 (dd, *J* = 8.3, 6.8 Hz, 1H), 3.82 (s, 3H), 1.78 – 1.61 (m, 6H), 1.60 – 1.52 (m, 1H), 1.37 – 1.28 (m, 1H), 1.26 – 1.11 (m, 3H), 0.99 – 0.86 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.5, 132.2, 128.3, 114.2, 63.5, 55.4, 43.6, 34.5, 33.5, 33.1, 26.6, 26.3, 26.2. IR (cm^{-1}): 2924, 2851, 2095, 1513, 1249, 829. HRMS: calcd. for $C_{15}H_{21}NO^+$ $[M–N_2]^+$: 231.1618, Found: 231.1577.

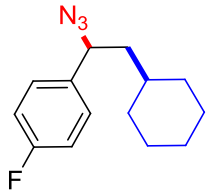
(3e) 1-(1-azido-2-cyclohexylethyl)-4-(chloromethyl)benzene



The title compound was prepared according to the general procedure described above by the reaction of 1-(chloromethyl)-4-ethenyl-benzene (**1e**) with $TMSN_3$ and cyclohexane (**2a**), and purified by flash column chromatography as colorless oil (31.0 mg, 56%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 – 7.37 (m, 2H), 7.32 – 7.27 (m, 2H), 4.59 (s, 2H), 4.51 (dd, *J* = 8.3, 6.6 Hz, 1H), 1.79 – 1.62 (m, 6H), 1.59 – 1.51 (m, 1H), 1.38 – 1.31 (m, 1H), 1.27 – 1.11 (m, 3H), 1.00 – 0.87 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.6, 137.4, 129.2, 127.4, 63.5, 46.0, 43.9, 34.4, 33.5, 33.0, 26.6, 26.2, 26.1. IR (cm^{-1}): 2924, 2851, 2097, 1447, 1265, 684. HRMS: calcd. for $C_{15}H_{21}ClN^+$ $[M–N_2+H]^+$: 250.1357, Found: 250.1341.

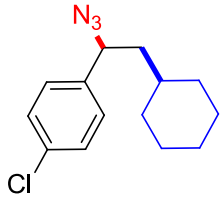
(3f) 1-(1-azido-2-cyclohexylethyl)-4-fluorobenzene



The title compound was prepared according to the general procedure described above by the reaction of 4-fluorostyrene (**1f**) with $TMSN_3$ and cyclohexane (**2a**), and purified by flash column chromatography as colorless oil (36.0 mg, 73%).

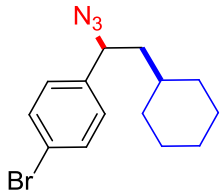
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.25 (m, 2H), 7.10 – 7.03 (m, 2H), 4.50 (dd, *J* = 8.5, 6.6 Hz, 1H), 1.78 – 1.61 (m, 6H), 1.56 – 1.50 (m, 1H), 1.38 – 1.27 (m, 1H), 1.26 – 1.08 (m, 3H), 1.01 – 0.87 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.6 (d, *J* = 245.2 Hz), 136.1 (d, *J* = 3.3 Hz), 128.7 (d, *J* = 8.1 Hz), 115.8 (d, *J* = 21.4 Hz). 63.2, 43.9, 34.4, 33.5, 33.0, 26.6, 26.2, 26.2. IR (cm^{-1}): 2924, 2852, 2097, 1510, 1231, 833. HRMS: calcd. for $C_{14}H_{19}FN^+$ $[M–N_2+H]^+$: 220.1496, Found: 220.1474.

(3g) 1-(1-azido-2-cyclohexylethyl)-4-chlorobenzene



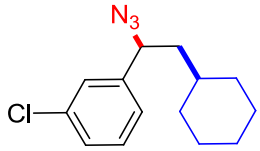
The title compound was prepared according to the general procedure described above by the reaction of 4-chlorostyrene (**1g**) with $TMSN_3$ and cyclohexane (**2a**), and purified by flash column chromatography as colorless oil (36.5 mg, 68%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.33 (m, 2H), 7.28 – 7.20 (m, 2H), 4.49 (dd, *J* = 8.8, 6.4 Hz, 1H), 1.77 – 1.62 (m, 6H), 1.56 – 1.48 (m, 1H), 1.38 – 1.27 (m, 1H), 1.26 – 1.10 (m, 3H), 1.00 – 0.87 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.8, 134.1, 129.1, 128.4, 63.3, 43.9, 34.4, 33.5, 33.0, 26.6, 26.2, 26.2. IR (cm^{-1}): 2924, 2851, 2096, 1448, 1240, 818. HRMS: calcd. for $C_{14}H_{19}ClN^+$ $[M–N_2+H]^+$: 236.1201, Found: 236.1188.

(3h) 1-(1-azido-2-cyclohexylethyl)-4-bromobenzene



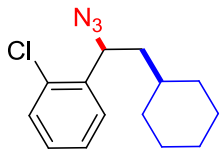
The title compound was prepared according to the general procedure described above by the reaction of 4-bromostyrene (**1h**) with $TMSN_3$ and cyclohexane (**2a**), and purified by flash column chromatography as colorless oil (36.8 mg, 60%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 – 7.47 (m, 2H), 7.20 – 7.15 (m, 2H), 4.48 (dd, *J* = 8.4, 6.4 Hz, 1H), 1.80 – 1.60 (m, 6H), 1.56 – 1.48 (m, 1H), 1.37– 1.27 (m, 1H), 1.26 – 1.10 (m, 3H), 1.00 – 0.86 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.4, 132.1, 128.7, 122.2, 63.3, 43.8, 34.4, 33.5, 33.0, 26.5, 26.2, 26.1. IR (cm^{-1}): 2923, 2851, 2096, 1488, 1240, 821. HRMS: calcd. for $C_{14}H_{19}BrN^+$ $[M–N_2+H]^+$: 280.0695, Found:280.0660.

(3i) 1-(1-azido-2-cyclohexylethyl)-3-chlorobenzene



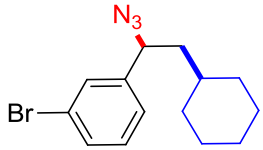
The title compound was prepared according to the general procedure described above by the reaction of 3-chlorostyrene (**1i**) with $TMSN_3$ and cyclohexane (**2a**), and purified by flash column chromatography as colorless oil (36.8 mg, 70%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 – 7.27 (m, 3H), 7.20 – 7.16 (m, 1H), 4.48 (dd, *J* = 8.6, 6.2 Hz, 1H), 1.80 – 1.63 (m, 6H), 1.57 – 1.48 (m, 1H), 1.40 – 1.30 (m, 1H), 1.27 – 1.110 (m, 3H), 1.00 – 0.86 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.5, 134.8, 130.2, 128.5, 127.1, 125.1, 63.4, 43.9, 34.4, 33.6, 32.9, 26.6, 26.2, 26.1. IR (cm^{-1}): 2924, 2851, 2097, 1448, 1248, 699. HRMS: calcd. for $C_{14}H_{19}ClN^+$ $[M–N_2+H]^+$: 236.1201, Found: 236.1189..

(3j) 1-(1-azido-2-cyclohexylethyl)-2-chlorobenzene



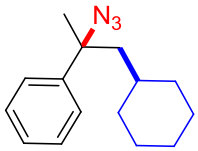
The title compound was prepared according to the general procedure described above by the reaction of 2-chlorostyrene (**1j**) with $TMSN_3$ and cyclohexane (**2a**), and purified by flash column chromatography as colorless oil (39.9 mg, 76%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 – 7.43 (m, 1H), 7.38 (dd, *J* = 8.0, 0.8 Hz, 1H), 7.35 – 7.28 (m, 1H), 7.26 – 7.21 (m, 1H), 5.10 (dd, *J* = 9.2, 4.8 Hz, 1H), 1.89 (d, *J* = 12.3 Hz, 1H), 1.78 – 1.56 (m, 6H), 1.51 – 1.40 (m, 1H), 1.30 – 1.13 (m, 3H), 1.02 – 0.91 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.5, 132.9, 129.9, 129.1, 127.7, 127.5, 60.0, 43.4, 34.7, 33.9, 32.5, 26.6, 26.3, 26.1. IR (cm^{-1}): 2923, 2851, 2099, 1447, 1249, 754. HRMS: calcd. for $C_{14}H_{19}ClN^+$ $[M–N_2+H]^+$: 236.1201, Found: 236.1188.

(3k) 1-(1-azido-2-cyclohexylethyl)-3-bromobenzene



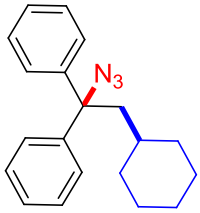
The title compound was prepared according to the general procedure described above by the reaction between 3-bromostyrene (**1k**) with $TMSN_3$ and cyclohexane (**2a**), and purified by flash column chromatography as colorless oil (44.8 mg, 73%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 – 7.42 (m, 2H), 7.26 – 7.21 (m, 2H), 4.47 (dd, *J* = 8.8, 6.0 Hz, 1H), 1.81 – 1.62 (m, 6H), 1.56 – 1.48 (m, 1H), 1.42 – 1.30 (m, 1H), 1.28 – 1.12 (m, 3H), 1.00 – 0.86 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.8, 131.4, 130.5, 130.0, 125.6, 123.0, 63.3, 43.9, 34.4, 33.6, 32.9, 26.5, 26.2, 26.1. IR (cm^{-1}): 2924, 2851, 2094, 1448, 1246, 697. HRMS: calcd. for $C_{14}H_{19}BrN^+$ $[M–N_2+H]^+$: 280.0695, Found:280.0661.

(3l) (2-azido-1-cyclohexylpropan-2-yl)benzene



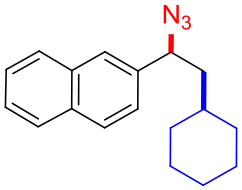
The title compound was prepared according to the general procedure described above by the reaction of alpha-methylstyrene (**1l**) with $TMSN_3$ and cyclohexane (**2a**), and purified by flash column chromatography as colorless oil (35.0 mg, 72%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.32 (m, 4H), 7.28 – 7.23 (m, 1H), 1.77 – 1.73 (m, 2H), 1.66 (s, 3H), 1.65 – 1.51 (m, 4H), 1.44 – 1.38 (m, 1H), 1.31 – 1.20 (m, 1H), 1.14 – 1.04 (m, 3H), 0.96 – 0.78 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.3, 128.9, 128.3, 127.0, 64.6, 44.4, 35.9, 34.8, 34.1, 28.6, 26.3, 26.2. IR (cm^{-1}): 2959, 2929, 2095, 1454, 1244, 699. HRMS: calcd. for $C_{15}H_{22}N^+$ $[M–N_2+H]^+$: 216.1747, Found: 216.1731.

(3m) (1-azido-2-cyclohexylethane-1,1-diyl)dibenzene



The title compound was prepared according to the general procedure described above by the reaction of 1,1-diphenylethylene (**1m**) with $TMSN_3$ and cyclohexane (**2a**), and purified by flash column chromatography as colorless oil (51.8 mg, 85%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.22 (m, 10H), 2.35 – 2.28 (m, 2H), 1.60 – 1.46 (m, 5H), 1.32 – 1.23 (m, 1H), 1.13 – 0.91 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.7, 128.3, 127.4, 127.2, 72.6, 45.6, 34.6, 33.7, 26.4, 26.3. IR (cm^{-1}): 2922, 2851, 2102, 1446, 1259, 698. HRMS: calcd. For $C_{20}H_{23}N^+$ $[M–N_2]^+$: 277.1825, Found: 277.1819.

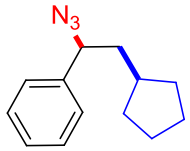
(3n) 2-(1-azido-2-cyclohexylethyl)naphthalene



The title compound was prepared according to the general procedure described above by the reaction of 2-vinylnaphthalene (**1n**) with $TMSN_3$ and cyclohexane (**2a**), and purified by flash column chromatography as colorless oil (34.6 mg, 62%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 – 7.82 (m, 3H), 7.34 (s, 1H), 7.52 – 7.49 (m, 2H), 7.43 (dd, J = 8.5, 1.7 Hz, 1H), 4.68 (dd, J = 8.5, 6.6 Hz, 1H), 1.85 – 1.74 (m, 3H), 1.72 – 1.61 (m, 4H), 1.43 – 1.32 (m, 1H), 1.26 – 1.10 (m, 3H), 1.01 – 0.88 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.6, 133.3, 133.3, 128.9, 128.1, 127.9, 126.5, 126.4, 126.2, 124.6, 64.2, 43.8, 34.5, 33.5, 33.1, 26.6, 26.2, 26.2. IR (cm^{-1}): 2923, 2850,

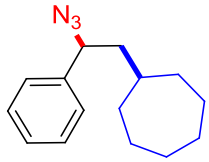
2097, 1447, 1240, 748. HRMS: calcd. for $C_{18}H_{22}N^+$ $[M–N_2+H]^+$: 252.1747, Found: 252.1724.

(3o) (1-azido-2-cyclopentylethyl)benzene



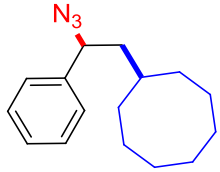
The title compound was prepared according to the general procedure described above by the reaction of styrene (**1a**) with $TMSN_3$ and cyclopentan (**2b**), and purified by flash column chromatography as colorless oil (31.2 mg, 72%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.30 (m, 5H), 4.43 (t, *J* = 7.0 Hz, 1H), 1.93 – 1.85 (m, 1H), 1.84 – 1.71 (m, 4H), 1.67 – 1.57 (m, 2H) , 1.56 – 1.46 (m, 2H), 1.19 – 1.07 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.1, 128.9, 128.3, 127.1, 65.9, 42.6, 37.0, 32.7, 25.2, 25.1. IR (cm^{-1}): 2949, 2867, 2560, 2094, 1246, 700. HRMS: calcd. for $C_{13}H_{18}N^+$ $[M+H–N_2]^+$: 188.1439, Found: 188.1434.

(3p) (2-azido-2-phenylethyl)cycloheptane



The title compound was prepared according to the general procedure described above by the reaction of styrene (**1a**) with $TMSN_3$ and cycloheptane (**2c**), and purified by flash column chromatography as colorless oil (34.0 mg, 70%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.28 (m, 5H), 4.47 (dd, *J* = 8.6, 5.8 Hz, 1H), 1.80 – 1.68 (m, 3H), 1.66 – 1.54 (m, 6H), 1.50 – 1.36 (m, 4H), 1.27 – 1.16 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.1, 128.5, 127.2, 125.6, 67.1, 49.3, 34.8, 34.5, 34.0, 26.8, 26.4, 26.3. IR (cm^{-1}): 2922, 2851, 2102, 1446, 1255, 698. HRMS: calcd. for $C_{15}H_{22}N^+$ $[M–N_2+H]^+$: 216.1747, Found: 216.1731.

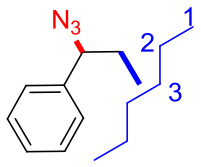
(3q) (2-azido-2-phenylethyl)cyclooctane



The title compound was prepared according to the general procedure described above by the reaction between styrene (**1a**) with $TMSN_3$ and cyclooctane (**2d**), and purified by flash column chromatography as colorless oil (37.5 mg, 73%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.28 (m, 5H), 4.48 (dd, *J* = 8.6, 5.4 Hz, 1H), 1.76 (t, *J* = 8.4 Hz, 1H), 1.69 – 1.53 (m, 9H), 1.49 – 1.39 (m, 5H), 1.35 – 1.27 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.4, 128.9, 128.3, 127.0, 64.6, 44.4, 33.9, 32.5, 31.6,

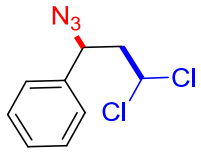
27.4, 27.4, 26.3, 25.4, 25.3. IR (cm^{-1}): 2922, 2852, 2094, 1448, 1243, 699. HRMS: calcd. for $C_{16}H_{23}N^+$ $[M–N_2]^+$: 229.1825, Found: 229.1805.

(3r) (1-azido-3-methylheptyl)benzene



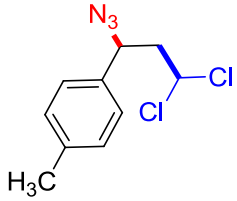
The title compound was prepared according to the general procedure described above by the reaction of styrene (**1a)** with $TMSN_3$ and n-hexane (**2e**), and purified by flash column chromatography as colorless oil (25.5 mg, 55%).
^1H NMR (400 MHz, $CDCl_3$) δ 7.43 – 7.37 (m, 15.5×1H), 7.37 – 7.30 (m, 22H), 4.55 – 4.47 (m, 6.5×1H), 4.42 (t, *J* = 7.2 Hz, 1H), 1.97 – 1.55 (m, 15H), 1.53 – 1.08 (m, 52.5×1H), 0.98 – 0.82 (m, 45H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.5, 140.4, 140.3, 140.1, 139.9, 128.9, 128.8, 128.3, 128.3 128.2, 127.1, 127.0, 127.0, 126.9, 66.5, 64.6, 64.4, 43.8, 43.2, 40.2, 36.9, 36.4, 36.3, 35.5, 35.48, 35.36, 35.1, 31.9, 29.8, 29.7, 29.3, 29.2, 29.1, 29.0, 26.3, 25.9, 25.4, 23.0, 22.7, 19.8, 19.6, 19.5, 19.4, 14.5, 14.2, 14.2, 10.6, 10.4. IR (cm^{-1}): 2957, 2928, 2094, 1454, 1243, 699. HRMS: calcd. For $C_{14}H_{21}N^+$ $[M–N_2]^+$: 203.1669, Found: 203.1627.

(4a) (1-azido-3,3-dichloropropyl)benzene



The title compound was prepared according to the general procedure described above by the reaction of styrene (**1a**) with $TMSN_3$ and dichloromethane, and purified by flash column chromatography as colorless oil (34.4 mg, 75%).
^1H NMR (400 MHz, $CDCl_3$) δ 7.46 – 7.32 (m, 5H), 5.74 (dd, *J* = 8.4, 4.9 Hz, 1H), 4.75 (dd, *J* = 9.5, 5.1 Hz, 1H), 2.65 (ddd, *J* = 14.4, 9.5, 4.9 Hz, 1H), 2.48 (ddd, *J* = 14.0, 8.4, 5.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.6, 129.4, 129.2, 127.1, 70.2, 63.3, 49.8. IR (cm^{-1}): 2103, 1455, 1249, 759, 700. HRMS: calcd. for $C_9H_9Cl_2N^+$ $[M–N_2]^+$: 201.0107, Found: 201.0086.

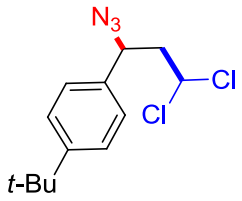
(4b) 1-(1-azido-3,3-dichloropropyl)-4-methylbenzene



The title compound was prepared according to the general procedure described above by the reaction of 4-methylphenylene (**1b**) with $TMSN_3$ and dichloromethane, and purified by flash column chromatography as colorless oil (34.5 mg, 71%).

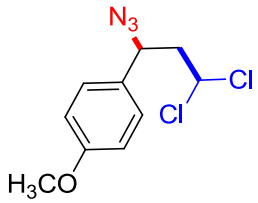
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 (s, 4H), 5.71 (dd, *J* = 8.2, 5.0 Hz, 1H), 4.71 (dd, *J* = 9.4, 5.2 Hz, 1H), 2.64 (ddd, *J* = 14.4, 9.4, 5.1 Hz, 1H), 2.46 (ddd, *J* = 14.4, 8.4, 5.2 Hz, 1H), 2.37 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.1, 134.5, 130.0, 127.0, 70.3, 63.1, 49.8, 21.3. IR (cm^{-1}): 2960, 2110, 1243, 818, 748. HRMS: calcd. for $C_{10}H_{11}Cl_2N^+$ [M–N_2]$^+$: 215.0263, Found: 215.0193.

(4c) 1-(1-azido-3,3-dichloropropyl)-4-(*tert*-butyl)benzene



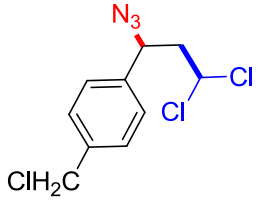
The title compound was prepared according to the general procedure described above by the reaction of 4-*tert*-butylstyrene (**1c**) with $TMSN_3$ and dichloromethane, and purified by flash column chromatography as colorless oil (46.7mg, 82%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 – 7.41 (m, 2H), 7.28 – 7.24 (m, 2H), 5.73 (dd, *J* = 8.2, 5.0 Hz, 1H), 4.72 (dd, *J* = 9.4, 5.3 Hz, 1H), 2.64 (ddd, *J* = 14.4, 9.2, 5.0 Hz, 1H), 2.47 (ddd, *J* = 14.0, 8.2, 5.4 Hz, 1H), 1.33 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.3, 134.5, 126.8, 126.2, 70.3, 63.0, 49.8, 34.8, 31.4. IR (cm^{-1}): 2964, 2105, 1246, 572. HRMS: calcd. For $C_{13}H_{17}Cl_2N^+$ [M– N_2]$^+$: 257.0733, Found: 257.0699.

(4d) 1-(1-azido-3,3-dichloropropyl)-4-methoxybenzene



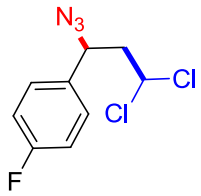
The title compound was prepared according to the general procedure described above by the reaction of 4-methoxystyrene (**1d**) with $TMSN_3$ and dichloromethane, and purified by flash column chromatography as colorless oil (22.2 mg, 43%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 – 7.24 (m, 2H), 6.96 – 6.90 (m, 2H), 5.70 (dd, *J* = 8.0, 5.2 Hz, 1H), 4.70 (dd, *J* = 9.2, 5.2 Hz, 1H), 3.83 (s, 3H), 2.64 (ddd, *J* = 14.4, 9.4, 5.2 Hz, 1H), 2.45 (ddd, *J* = 14.0, 8.2, 5.3 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.2, 129.4, 128.4, 114.8, 70.3, 62.8, 55.5, 49.8. IR (cm^{-1}): 2104, 1514, 1611, 1251, 833. HRMS: calcd. for $C_{10}H_{11}Cl_2NO^+$ [M–N_2]$^+$: 231.0212, Found: 231.0182.

(4e) 1-(1-azido-3,3-dichloropropyl)-4-(chloromethyl)benzene



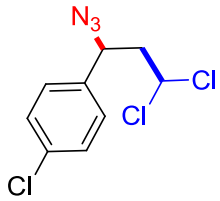
The title compound was prepared according to the general procedure described above by the reaction of 1-(chloromethyl)-4-ethenyl-benzene (**1e**) with $TMSN_3$ and dichloromethane, and purified by flash column chromatography as colorless oil (35.3 mg, 64%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 – 7.43 (m, 2H), 7.37 – 7.32 (m, 2H), 5.74 (dd, *J* = 8.4, 4.8 Hz, 1H), 4.76 (dd, *J* = 9.6, 5.2 Hz, 1H), 4.60 (s, 2H), 2.63 (ddd, *J* = 14.4, 9.6, 4.8 Hz, 1H), 2.46 (ddd, *J* = 13.6, 8.6, 5.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.1, 137.9, 129.6, 127.5, 70.0, 63.0, 49.8, 45.6. IR (cm^{-1}): 2360, 2105, 1246, 682. EI MS m/z: $C_{10}H_{10}Cl_3^+$ $[M–N_3]^+$: 235.0.

(4f) 1-(1-azido-3,3-dichloropropyl)-4-fluorobenzene



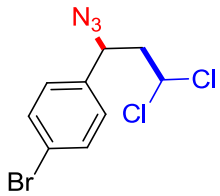
The title compound was prepared according to the general procedure described above by the reaction between 4-fluorostyrene (**1f**) with $TMSN_3$ and dichloromethane, and purified by flash column chromatography as colorless oil (34.5 mg, 70%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.30 (m, 2H), 7.15 – 7.09 (m, 2H), 5.74 (dd, *J* = 8.2, 5.0 Hz, 1H), 4.75 (dd, *J* = 9.4, 5.2 Hz, 1H), 2.63 (ddd, *J* = 14.4, 9.4, 5.2 Hz, 1H), 2.45 (ddd, *J* = 13.8, 8.4, 5.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.0 (d, *J* = 248.3 Hz), 133.5 (d, *J* = 3.3 Hz), 128.9 (d, *J* = 8.3 Hz), 116.4 (d, *J* = 21.8 Hz), 70.0, 62.6, 49.9. IR (cm^{-1}): 2360, 2106, 1510, 1229, 836. EI MS m/z: $C_9H_8Cl_2F^+$ $[M–N_3]^+$: 205.0.

(4g) 1-(1-azido-3,3-dichloropropyl)-4-chlorobenzene



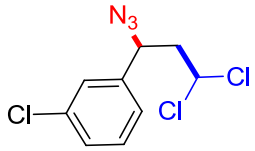
The title compound was prepared according to the general procedure described above by the reaction of 4-chlorostyrene (**1g**) with $TMSN_3$ and dichloromethane, and purified by flash column chromatography as colorless oil (37.8 mg, 72%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 – 7.38 (m, 2H), 7.30 – 7.26 (m, 2H), 5.73 (dd, *J* = 8.4, 4.8 Hz, 1H), 4.74 (dd, *J* = 9.4, 5.0 Hz, 1H), 2.61 (ddd, *J* = 14.4, 9.6, 4.8 Hz, 1H), 2.45 (ddd, *J* = 14.0, 8.6, 5.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.2, 135.1, 129.6, 128.4, 69.9, 62.7, 49.8. IR (cm^{-1}): 2104, 1493, 1245, 1092, 829. HRMS: calcd. for $C_9H_8Cl_3N^+$ $[M–N_2]^+$: 234.9717, Found: 234.9659.

(4h) 1-(1-azido-3,3-dichloropropyl)-4-bromobenzene



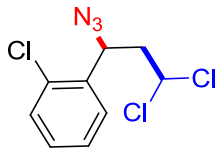
The title compound was prepared according to the general procedure described above by the reaction of 4-bromostyrene (**1h**) with $TMSN_3$ and dichloromethane, and purified by flash column chromatography as colorless oil (39.9 mg, 65%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 – 7.38 (m, 2H), 7.32 – 7.27 (m, 2H), 5.73 (dd, *J* = 8.4, 4.8 Hz, 1H), 4.74 (dd, *J* = 9.4, 5.2 Hz, 1H), 2.61 (ddd, *J* = 14.4, 9.6, 4.8 Hz, 1H), 2.44 (ddd, *J* = 14.4, 8.6, 5.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.2, 135.1, 129.6, 128.4, 69.9, 62.7, 49.8. IR (cm^{-1}): 2360, 2101, 1492, 1242, 827. HRMS: calcd. for $C_9H_8BrCl_2N^+$ $[M–N_2]^+$: 278.9212, Found: 278.9278.

(4i) 1-(1-azido-3,3-dichloropropyl)-3-chlorobenzene



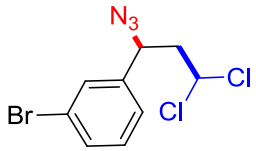
The title compound was prepared according to the general procedure described above by the reaction of 3-chlorostyrene (**1i**) with $TMSN_3$ and dichloromethane, and purified by flash column chromatography as colorless oil (38.4 mg, 73%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 – 7.41 (m, 2H), 7.38 – 7.28 (m, 2H), 5.90 (dd, *J* = 9.4, 3.8 Hz, 1H), 5.34 (dd, *J* = 10.2, 3.8 Hz, 1H), 2.56 (ddd, *J* = 14.4, 9.4, 4.0 Hz, 1H), 2.46 (ddd, *J* = 14.4, 10.0, 3.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.9, 135.4, 130.7, 129.4, 127.2, 125.2, 69.9, 62.8, 49.8. IR (cm^{-1}): 2361, 2109, 1477, 1245, 696. HRMS: calcd. for $C_9H_8Cl_3N^+$ $[M–N_2]^+$: 234.9717, Found: 234.9659.

(4j) 1-(1-azido-3,3-dichloropropyl)-2-chlorobenzene



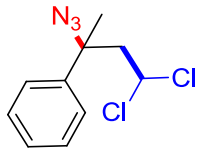
The title compound was prepared according to the general procedure described above by the reaction of 2-chlorostyrene (**1j**) with $TMSN_3$ and dichloromethane, and purified by flash column chromatography as colorless oil (37.9 mg, 72%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.33 (m, 3H), 7.26 – 7.21 (m, 1H), 5.75 (dd, *J* = 8.6, 4.8 Hz, 1H), 4.74 (dd, *J* = 9.7, 4.9 Hz, 1H), 2.60 (ddd, *J* = 14.4, 9.7, 4.7 Hz, 1H), 2.45 (ddd, *J* = 14.4, 8.6, 4.9 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 135.9, 133.0, 130.3, 130.0, 127.9, 127.8, 70.1, 59.9, 48.8. IR (cm^{-1}): 2363, 2112, 1479, 1249, 699. HRMS: calcd. for $C_9H_8Cl_3N^+$ $[M–N_2]^+$: 234.9717, Found: 234.9659.

(4k) 1-(1-azido-3,3-dichloropropyl)-3-bromobenzene



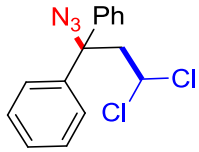
The title compound was prepared according to the general procedure described above by the reaction between 3-bromostyrene (**1k**) with $TMSN_3$ and dichloromethane, and purified by flash column chromatography as colorless oil (38.1 mg, 62%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 – 7.49 (m, 2H), 7.33 – 7.25 (m, 2H), 5.76 (dd, *J* = 8.8, 4.8 Hz, 1H), 4.73 (dd, *J* = 9.7, 4.9 Hz, 1H), 2.60 (ddd, *J* = 14.4, 9.7, 4.7 Hz, 1H), 2.45 (ddd, *J* = 14.4, 8.7, 4.9 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.1, 132.4, 130.9, 130.1, 125.8, 123.4, 69.9, 62.8, 49.9. IR (cm^{-1}): 2923, 2360, 2100, 1475, 1244, 697. HRMS: calcd. for $C_9H_8BrCl_2N^+$ $[M–N_2]^+$: 278.9212, Found: 278.9277.

(4l) (2-azido-4,4-dichlorobutan-2-yl)benzene



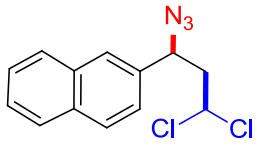
The title compound was prepared according to the general procedure described above by the reaction of alpha-methylstyrene (**1l**) with $TMSN_3$ and dichloromethane, and purified by flash column chromatography as colorless oil (38.4 mg, 79%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (m, 4H), 7.33 (m, 1H), 5.58 – 5.53 (m, 1H), 2.79 (dd, *J* = 5.6, 4.4 Hz, 2H), 1.82 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.7, 129.1, 128.2, 125.6, 69.0, 65.5, 55.4, 25.8. IR (cm^{-1}): 2359, 2110, 1446, 1250, 698. HRMS: calcd. for $C_{10}H_{11}C_{l2}N^+$ $[M–N_2]^+$: 215.0263, Found: 215.0193.

(4m) (1-azido-3,3-dichloropropane-1,1-diyl)dibenzene



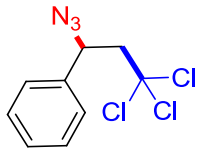
The title compound was prepared according to the general procedure described above by the reaction of 1,1-diphenylethylene (**1m**) with $TMSN_3$ and dichloromethane, and purified by flash column chromatography as colorless oil (49.1 mg, 81%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.28 (m, 10H), 5.43 (t, *J* = 5.4 Hz, 1H), 3.41 (d, *J* = 5.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.3, 128.9, 128.4, 127.0, 70.8, 69.0, 52.2. IR (cm^{-1}): 2112, 1447, 1257, 1000, 698. HRMS: calcd. for $C_{15}H_{13}Cl_2N^+$ $[M–N_2]^+$: 277.0420, Found: 277.0486.

(4n) 2-(1-azido-3,3-dichloropropyl)naphthalene



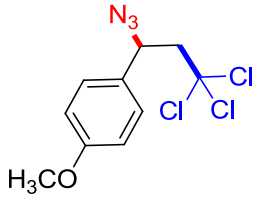
The title compound was prepared according to the general procedure described above by the reaction of 2-vinylnaphthalene (**1n**) with $TMSN_3$ and dichloromethane, and purified by flash column chromatography as colorless oil (34.6 mg, 62%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 – 7.84 (m, 3H), 7.80 (d, *J* = 1.2 Hz, 1H), 7.55 – 7.51 (m, 2H), 7.43 (dd, *J* = 8.5, 1.8 Hz, 1H), 5.76 (dd, *J* = 8.2, 5.0 Hz, 1H), 4.92 (dd, *J* = 9.3, 5.3 Hz, 1H), 2.73 (ddd, *J* = 14.4, 9.2, 5.2 Hz, 1H), 2.56 (ddd, *J* = 14.2, 8.2, 5.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 134.9, 133.6, 133.3, 129.6, 128.2, 128.0, 126.9, 126.9, 126.7, 124.1, 70.2, 63.6, 49.8. IR (cm^{-1}): 2110, 1243, 818, 748. HRMS: calcd. For $C_{13}H_{11}Cl_2N^+$ $[M–N_2]^+$: 251.0263, Found: 251.0211.

(5a) (1-azido-3,3,3-trichloropropyl)benzene [1]



The title compound was prepared according to the general procedure described above by the reaction of styrene (**1a**) with $TMSN_3$ and chloroform, and purified by flash column chromatography as colorless oil (32.2 mg, 67%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 – 7.34 (m, 5H), 4.95 (dd, *J* = 7.2, 3.9 Hz, 1H), 3.22 (dd, *J* = 15.2, 7.3 Hz, 1H), 3.11 (dd, *J* = 15.2, 3.9 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.8, 129.3, 129.0, 127.0, 96.8, 63.5, 60.2. IR (cm^{-1}): 2962, 2103, 1266, 1005, 700.

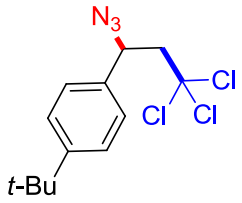
(5b) 1-(1-azido-3,3,3-trichloropropyl)-4-methoxybenzene



The title compound was prepared according to the general procedure described above by the reaction of 4-methoxystyrene (**1d**) with $TMSN_3$ and trichloromethane, and purified by flash column chromatography as colorless oil (34.6 mg, 59%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 – 7.26 (m, 2H), 6.97 – 6.91 (m, 2H), 4.91 (dd, *J* = 6.8, 4.5 Hz, 1H), 3.83 (s, 3H), 3.20 (dd, *J* = 15.2, 6.8 Hz, 1H), 3.09 (dd, *J* = 15.2, 4.4 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.0, 130.6, 128.4, 114.5, 96.8, 63.0, 60.0, 55.4. IR (cm^{-1}): 2105, 1519, 1615, 1253, 839. HRMS: calcd. for $C_{10}H_{11}Cl_3NO^+$ $[M–N_2+H]^+$: 265.9901, Found: 265.9895.

(5c)1-(1-azido-3,3,3-trichloropropyl)-4-(*tert*-butyl)benzene

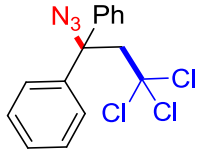
[1] B. Chen, C. Fang, P. Liu, J. M. Ready. *Angew. Chem. Int. Ed*. **2017**, *56*, 8780.



The title compound was prepared according to the general procedure described above by the reaction of 4-*tert*-butylstyrene (**1c**) with $TMSN_3$ and tetrachloride, and purified by flash column chromatography as colorless oil (30.0 mg, 47%).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 – 7.40 (m, 2H), 7.30 – 7.26 (m, 2H), 4.92 (dd, *J* = 7.5, 3.5 Hz, 1H), 3.21 (dd, *J* = 15.0, 7.6 Hz, 1H), 3.10 (dd, *J* = 15.2, 3.5 Hz, 1H), 1.33 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.1, 135.8, 126.6, 126.2, 97.0, 63.3, 60.2, 34.8, 31.4. IR (cm^{-1}): 2964, 2101, 1268, 1009, 706. HRMS: calcd. for $C_{13}H_{16}Cl_3N^+$ $[M–N_2]^+$: 291.0343, Found: 291.0299.

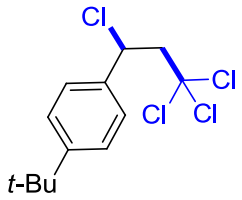
(5d) (1-azido-3,3,3-trichloropropane-1,1-diyl)dibenzene



The title compound was prepared according to the general procedure described above by the reaction of 1,1-diphenylethylene (**1m**) with $TMSN_3$ and tetrachloride, and purified by flash column chromatography as colorless oil (39.9 mg, 58%).

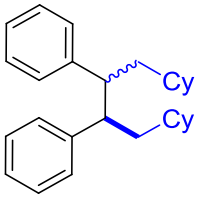
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.29 (m, 10H), 3.95 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.8, 128.6, 128.2, 127.3, 127.3, 95.8, 70.8, 59.4. IR (cm^{-1}): 2114, 1447, 1258, 1015, 697. HRMS: calcd. for $C_{15}H_{13}Cl_3N^+$ $[M–N_2+H]^+$: 312.0108, Found: 312.0997.

(6) 1-(*tert*-butyl)-4-(1,3,3,3-tetrachloropropyl)benzene



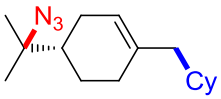
Colorless oil (9.5 mg, 15%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.33 (m, 4H), 5.29 (t, *J* = 5.8 Hz, 1H), 3.61 (dd, *J* = 15.4, 5.7 Hz, 1H), 3.52 (dd, *J* = 15.4, 5.9 Hz, 1H), 1.31 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.2, 137.6, 127.1, 126.0, 96.5, 62.9, 58.3, 34.8, 31.4. IR (cm^{-1}): 2969, 2108, 1279, 1010, 712. HRMS: calcd. for $C_{13}H_{17}Cl_4^+$ $[M+H]^+$: 313.0079, Found: 313.0073.

(7) 1,4-dicyclohexylbutane-2,3-diyl)dibenzene



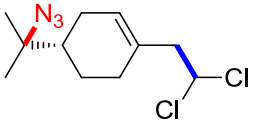
Melting point: 79 – 81.5 ℃.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 – 7.27 (m, 4H), 7.24 – 7.18 (m, 2H), 7.18 – 7.10 (m, 4H), 2.76 (dd, *J* = 6.3, 3.1 Hz, 2H), 1.70 (d, *J* = 12.0 Hz, 2H), 1.54 – 1.41 (m, 6H), 1.37 – 1.24 (m, 4H), 1.09 – 0.90 (m, 8H), 0.79 – 0.62 (m, 4H), 0.57 – 0.44 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.2, 128.5, 128.3, 126.0, 49.5, 42.5, 34.7, 34.6, 31.8, 26.7, 26.3, 26.1. IR (cm^{-1}): 2959, 2929, 1454, 1244, 699. HRMS: calcd. for $C_{28}H_{38}^{+}$ $[M]^{+}$: 274.2968, Found: 274.2999.

(9a) 4-(2-azidopropan-2-yl)-1-(cyclohexylmethyl)cyclohex-1-ene



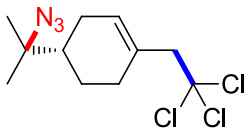
The title compound was prepared according to the general procedure described above by the reaction of ethyl beta-pinene (**8**) with $TMSN_3$ and cyclohexane (**2a**), and purified by flash column chromatography as colorless oil (38.6 mg, 74%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.33 (d, *J* = 4.6 Hz, 1H), 2.12 – 1.92 (m, 3H), 1.89 – 1.78 (m, 4H), 1.72 – 1.60 (m, 5H), 1.57 – 1.50 (m, 1H), 1.42 – 1.31 (m, 1H), 1.29 – 1.09 (m, 10H), 0.91 – 0.73 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.6, 121.1, 64.4, 45.9, 43.8, 35.6, 33.7, 33.2, 29.3, 26.9, 26.8, 26.5, 24.4, 24.0, 23.1. IR (cm^{-1}): 2960, 2334, 2108, 1224, 700. HRMS: calcd. for $C_{16}H_{27}N_3Na^{+}$ $[M+Na]^{+}$: 284.2097, Found: 284.2149.

(9b) 4-(2-azidopropan-2-yl)-1-(2,2-dichloroethyl)cyclohex-1-ene



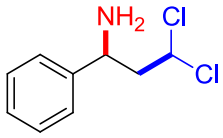
The title compound was prepared according to the general procedure described above by the reaction of beta-pinene (**8**) with $TMSN_3$ and dichloromethane, and purified by flash column chromatography as colorless oil (37.5 mg, 72%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.77 (t, *J* = 6.7 Hz, 1H), 5.61 (d, *J* = 2.2 Hz, 1H), 2.84 (d, *J* = 6.7 Hz, 2H), 2.20 – 1.99 (m, 3H), 1.94 – 1.84 (m, 2H), 1.56 (m, 1H), 1.34 – 1.27 (m, 1H), 1.27 (s, 3H), 1.24 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 132.2, 126.4, 72.0, 64.1, 51.5, 43.2, 29.1, 26.9, 24.0, 23.9, 23.2. IR (cm^{-1}): 2961, 2111, 1243, 699. HRMS: calcd. For $C_{11}H_{17}Cl_2N_3Na^{+}$ $[M+Na]^{+}$: 284.0692, Found: 284.0658.

(9c) 4-(2-azidopropan-2-yl)-1-(2,2,2-trichloroethyl)cyclohex-1-ene



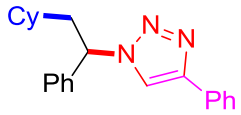
The title compound was prepared according to the general procedure described above by the reaction of beta-pinene (**8**) with $TMSN_3$ and trichloromethane, and purified by flash column chromatography as colorless oil (40 mg, 23.6%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 5.81 (s, 1H), 3.34 (s, 2H), 2.46 – 2.27 (m, 2H), 2.25 – 2.13 (m, 1H), 2.01 – 1.86 (m, 2H), 1.67 – 1.49 (m, 2H), 1.29 (s, 3H), 1.26 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 131.4, 130.8, 99.2, 64.1, 62.0, 43.1, 30.7, 27.3, 24.3, 23.9, 23.3. IR (cm^{-1}): 2964, 2110, 1238, 701. HRMS: calcd. For $C_{11}H_{16}Cl_3N_3Na^+$ $[M+Na]^+$: 318.0302, Found: 318.026

(14) 3,3-dichloro-1-phenylpropan-1-amine



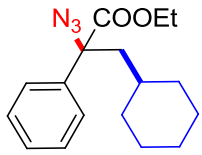
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.26 (m, 5H), 5.71 (t, *J* = 6.7 Hz, 1H), 4.80 – 3.88 (m, 1H), 2.58 – 2.44 (m, 2H), 1.51 (s, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 144.6, 129.0, 127.8, 126.2, 71.5, 53.7, 52.8. IR (cm^{-1}): 2960, 2443, 1459, 701. HRMS: calcd. for C9H11Cl_2NNa^+ $[M+Na]^+$: 226.0161, Found: 226.0156.

(15) 1-(2-cyclohexyl-1-phenylethyl)-4-phenyl-1H-1,2,3-triazole



Melting point: 137 - 140 ℃.
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 – 7.79 (m, 2H), 7.71 (s, 1H), 7.43 – 7.28 (m, 8H), 5.80 (dd, *J* = 9.2, 6.6 Hz, 1H), 2.48 – 2.36 (m, 1H), 2.13 (dt, *J* = 14.1, 7.0 Hz, 1H), 1.87 (d, *J* = 12.2 Hz, 1H), 1.80 – 1.60 (m, 4H), 1.21 – 1.11 (m, 4H), 1.07 – 0.95 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.8, 139.5, 130.8, 129.1, 128.9, 128.6, 128.2, 127.0, 125.8, 118.5, 62.7, 42.8, 34.2, 33.4, 32.9, 26.5, 26.1, 26.0.IR (cm^{-1}): 2923, 2851, 2103, 1446. HRMS: calcd. for $C_{22}H_{26}N_3^+$ $[M+H]^+$: 332.2121, Found: 332.2113.

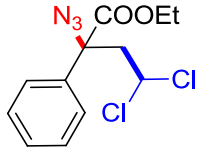
(11a) (1-azido-2-cyclohexylethyl)benzene



The title compound was prepared according to the general procedure described above by the reaction of ethyl 2-phenylacrylate (**10**) with $TMSN_3$ and cyclohexane (**2a**), and purified by flash column chromatography as colorless oil (42.1mg, 70%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 – 7.30 (m, 5H), 4.30 – 4.23 (m, 2H), 2.15 (dd, *J* = 14.5, 6.2 Hz, 1H), 2.02 (dd, *J* = 14.5, 5.3 Hz, 1H), 1.75 – 1.55 (m, 5H), 1.48 – 1.37 (m,

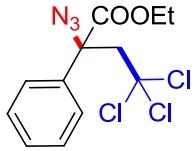
1H), 1.28 (t, *J* = 7.1 Hz, 3H), 1.22 – 1.07 (m, 3H), 1.04 – 0.89 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.5, 138.8, 128.8, 128.4, 126.0, 72.8, 62.3, 45.0, 34.7, 34.3, 33.8, 26.4, 26.2, 14.1. IR (cm^{-1}): 2956, 2359, 2118, 1735, 1445, 1208, 699. HRMS: calcd. for $C_{17}H_{23}N_3O_2Na^+$ $[M+Na]^+$: 324.1682, Found: 324.1682.

(11b) ethyl 2-azido-4,4-dichloro-2-phenylbutanoate



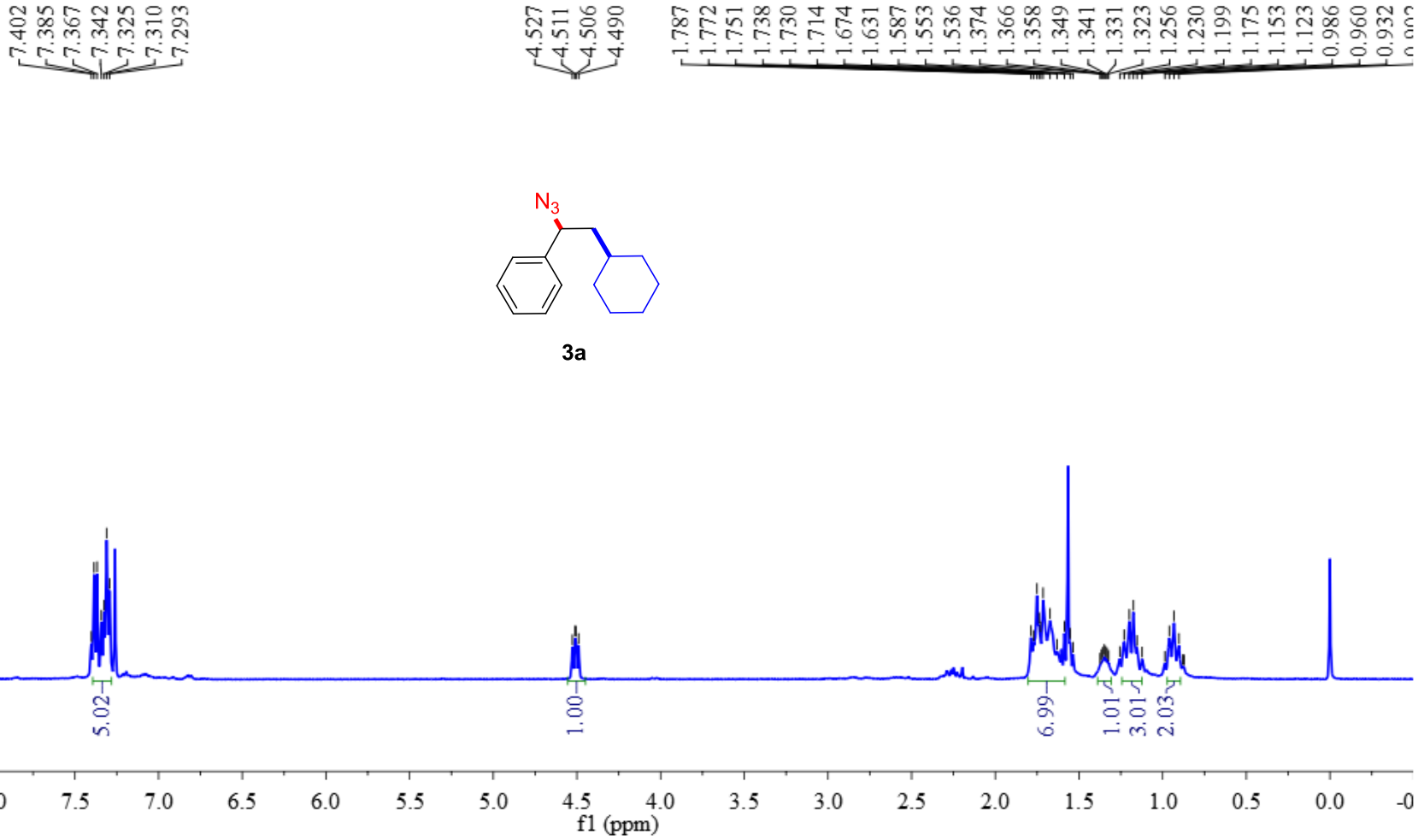
The title compound was prepared according to the general procedure described above by the reaction of 2-phenylacrylate (**10**) with $TMSN_3$ and dichloromethane, and purified by flash column chromatography as colorless oil (45.7 mg, 76%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 – 7.37 (m, 5H), 5.91 (dd, *J* = 8.8, 3.7 Hz, 1H), 4.40 – 4.28 (m, 2H), 3.23 (dd, *J* = 15.1, 8.8 Hz, 1H), 2.93 (dd, *J* = 15.1, 3.7 Hz, 1H), 1.34 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.1, 137.0, 129.4, 129.2, 125.9, 70.2, 68.8, 63.0, 51.0, 14.0. IR (cm^{-1}): 2960, 2360, 2110, 1736, 1448, 1201.699. HRMS: calcd. for $C_{12}H_{13}Cl_2N_3O_2Na^+$ $[M+Na]^+$: 324.0277, Found: 324.0256.

(11c) ethyl 2-azido-4,4,4-trichloro-2-phenylbutanoate

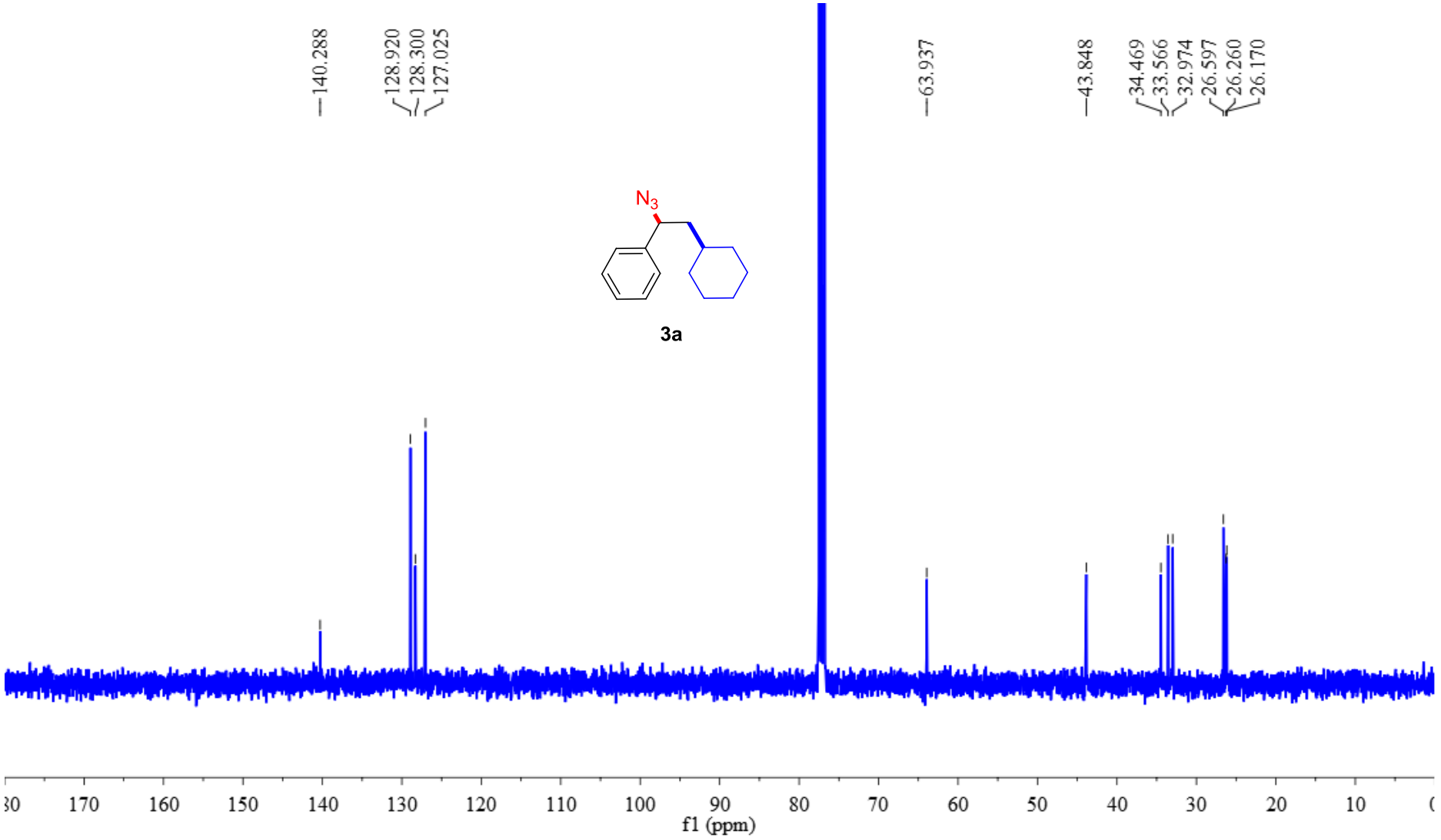


The title compound was prepared according to the general procedure described above by the reaction of 2-phenylacrylate (**10**) with $TMSN_3$ and trichloromethane, and purified by flash column chromatography as colorless oil (46.2 mg, 69%).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 – 7.37 (m, 5H), 4.43 – 4.27 (m, 2H), 4.02 (d, *J* = 15.5 Hz, 1H), 3.45 (d, *J* = 15.5 Hz, 1H), 1.33 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.9, 137.1, 129.4, 129.3, 126.1, 95.0, 71.0, 63.3, 59.5, 14.0. IR (cm^{-1}): 2960, 2101, 1737, 1266, 1004, 699. HRMS: calcd. For $C_{12}H_{12}Cl_3N_3O_2Na^+$ $[M+Na]^+$: 357.9887, Found: 357.9844.

VII. Copies of ^{1}H and ^{13}C NMR spectra of products



3a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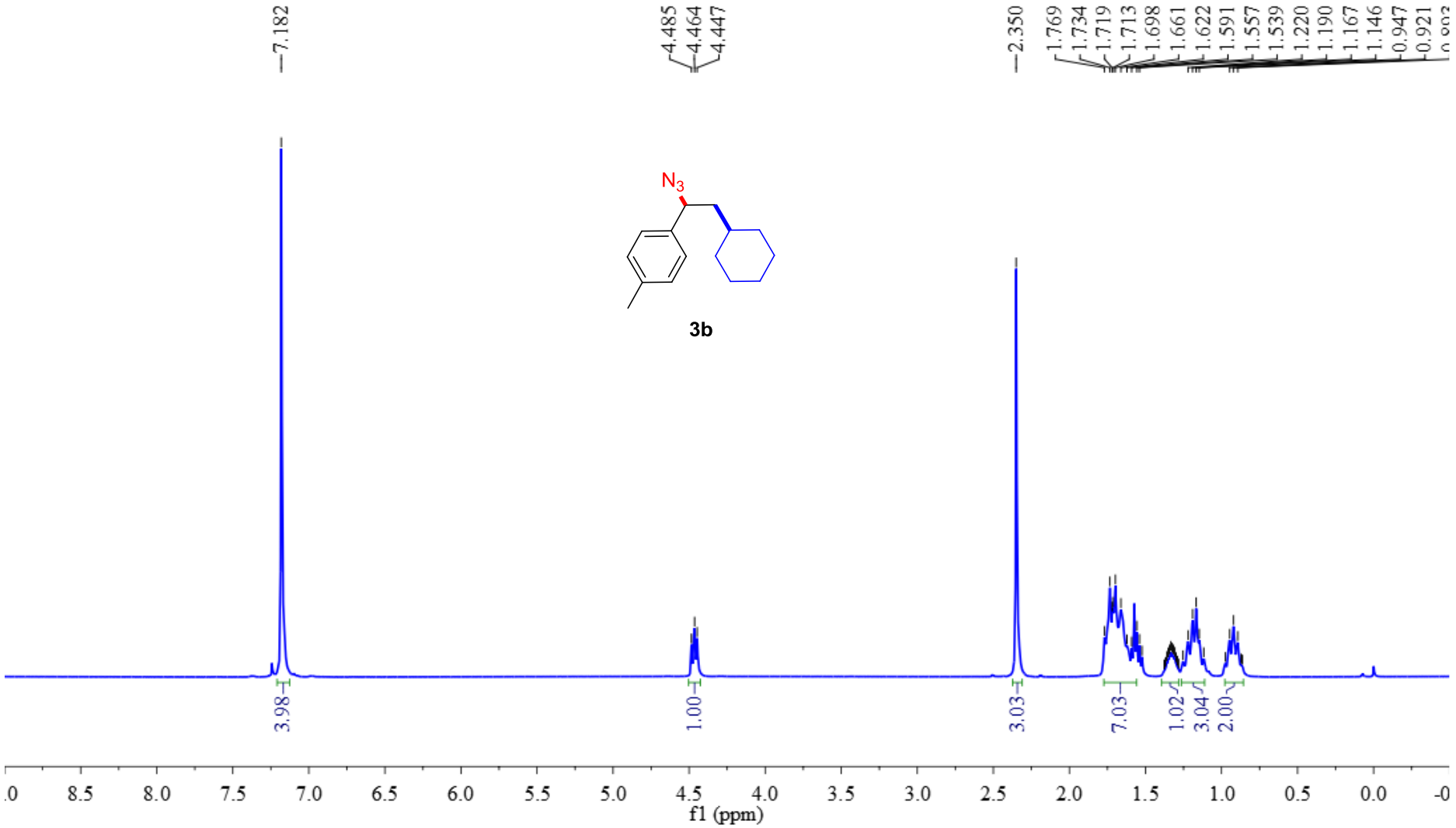
128.300
127.025
63.937
43.848
34.469
33.566
32.974
26.597
26.260
26.170
N3
3a
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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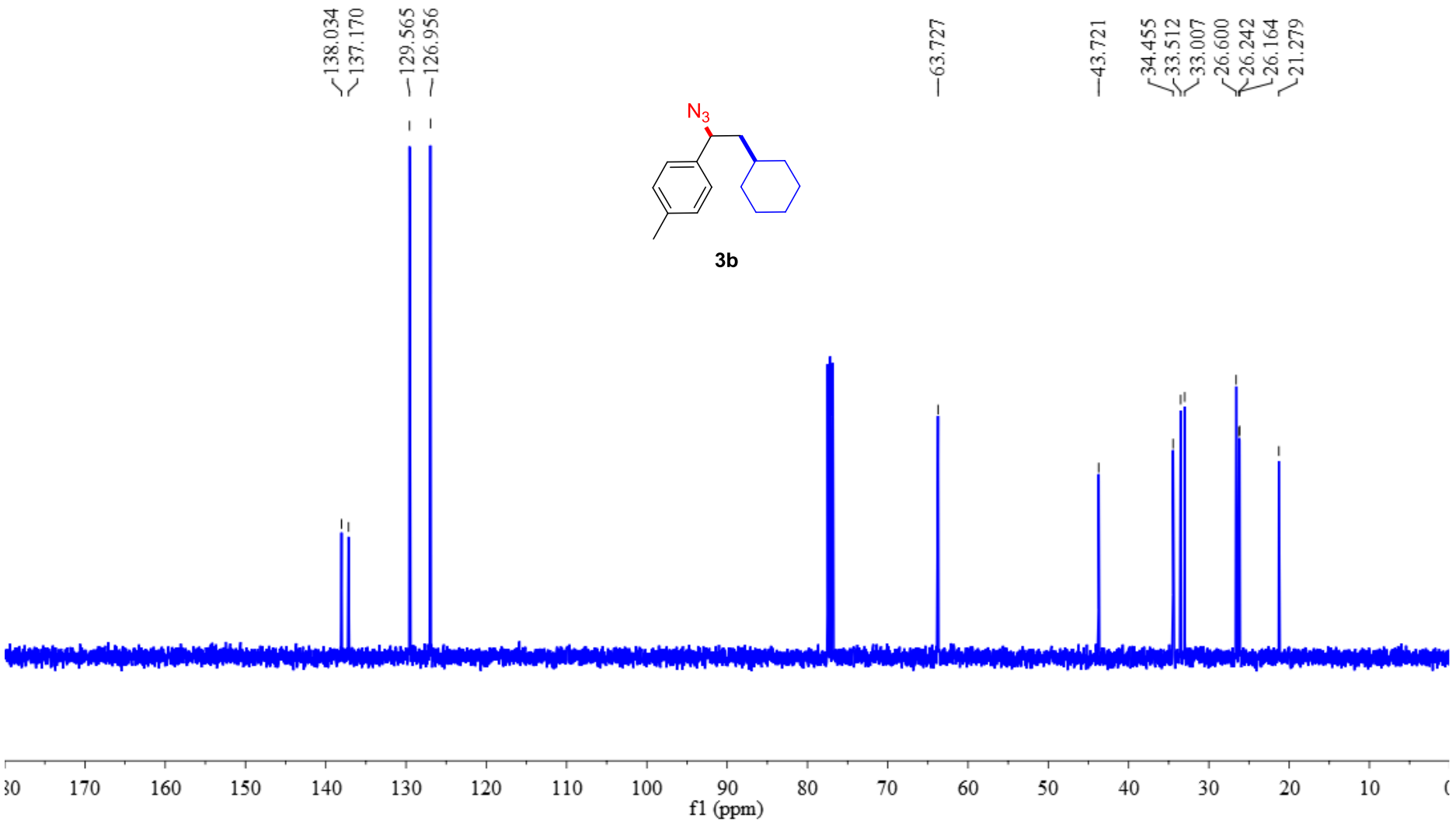
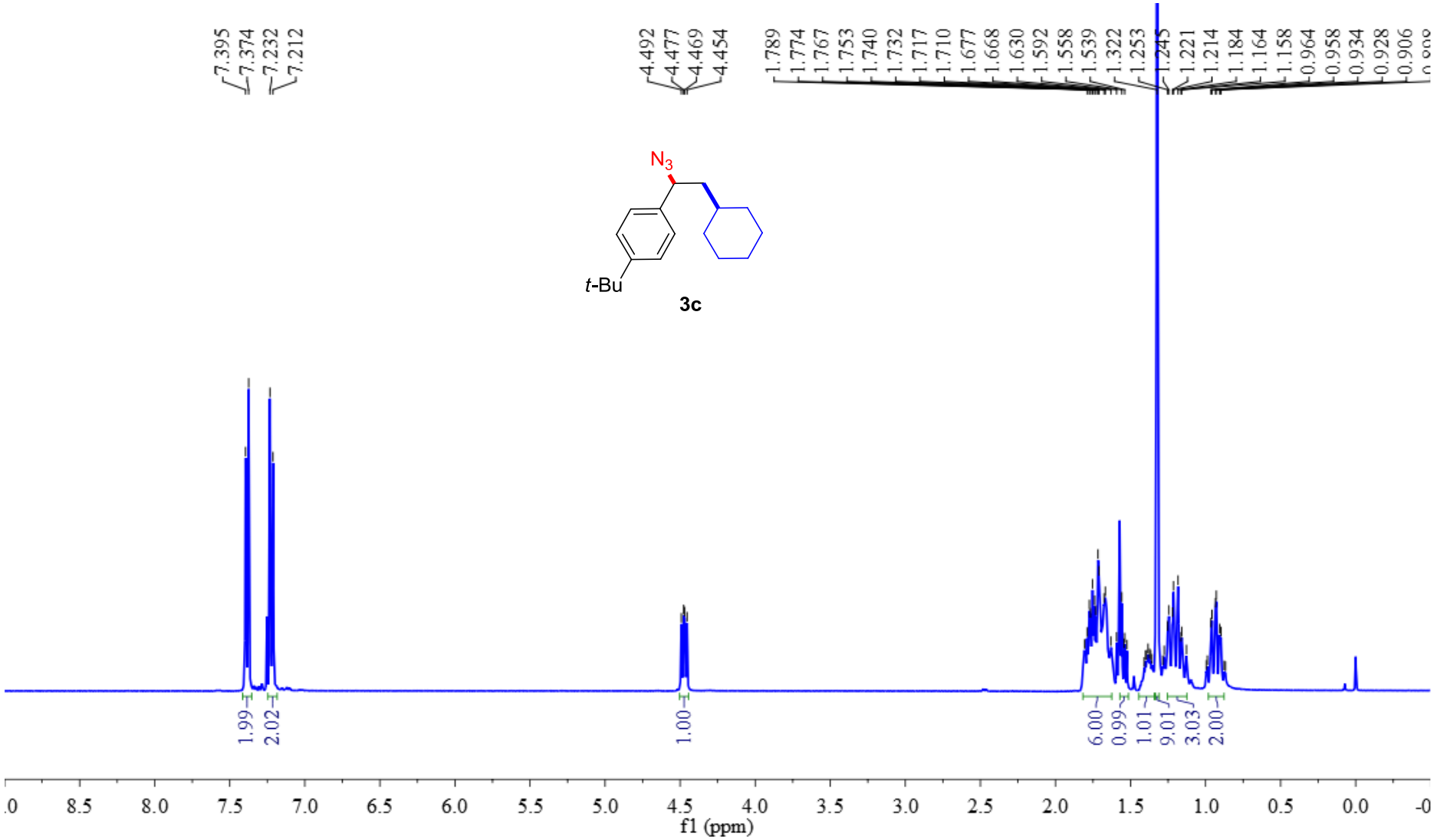
4.485
4.464
4.447
2.350
1.769
1.734
1.719
1.713
1.698
1.661
1.622
1.591
1.557
1.539
1.220
1.190
1.167
1.146
0.947
0.921
N3
3b
3.98
1.00
3.03
7.03
1.02
3.04
2.00
f1 (ppm)


138.034
137.170
129.565
126.956
63.727
43.721
34.455
33.512
33.007
26.600
26.242
26.164
21.279
N3
3b
f1 (ppm)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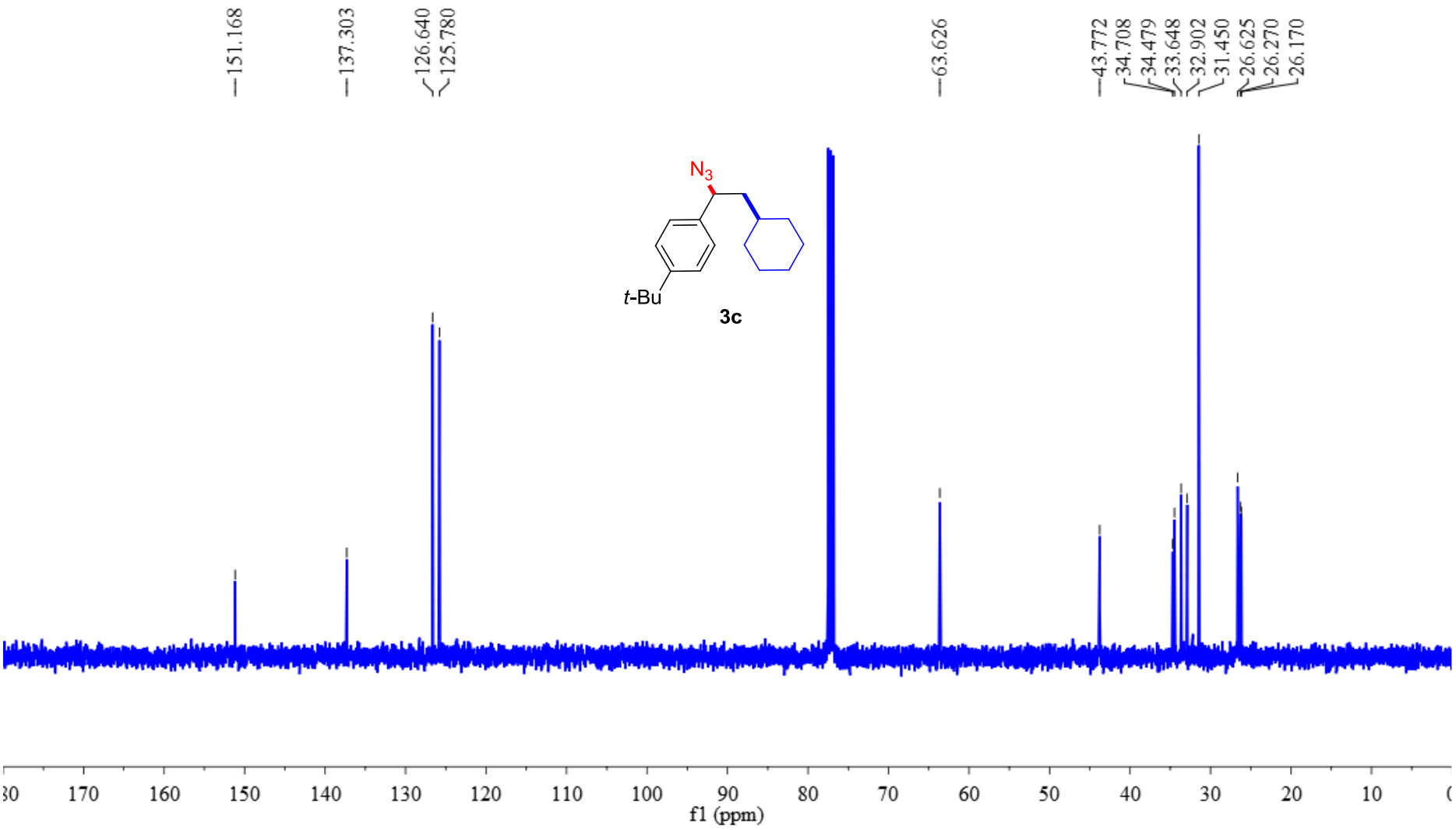
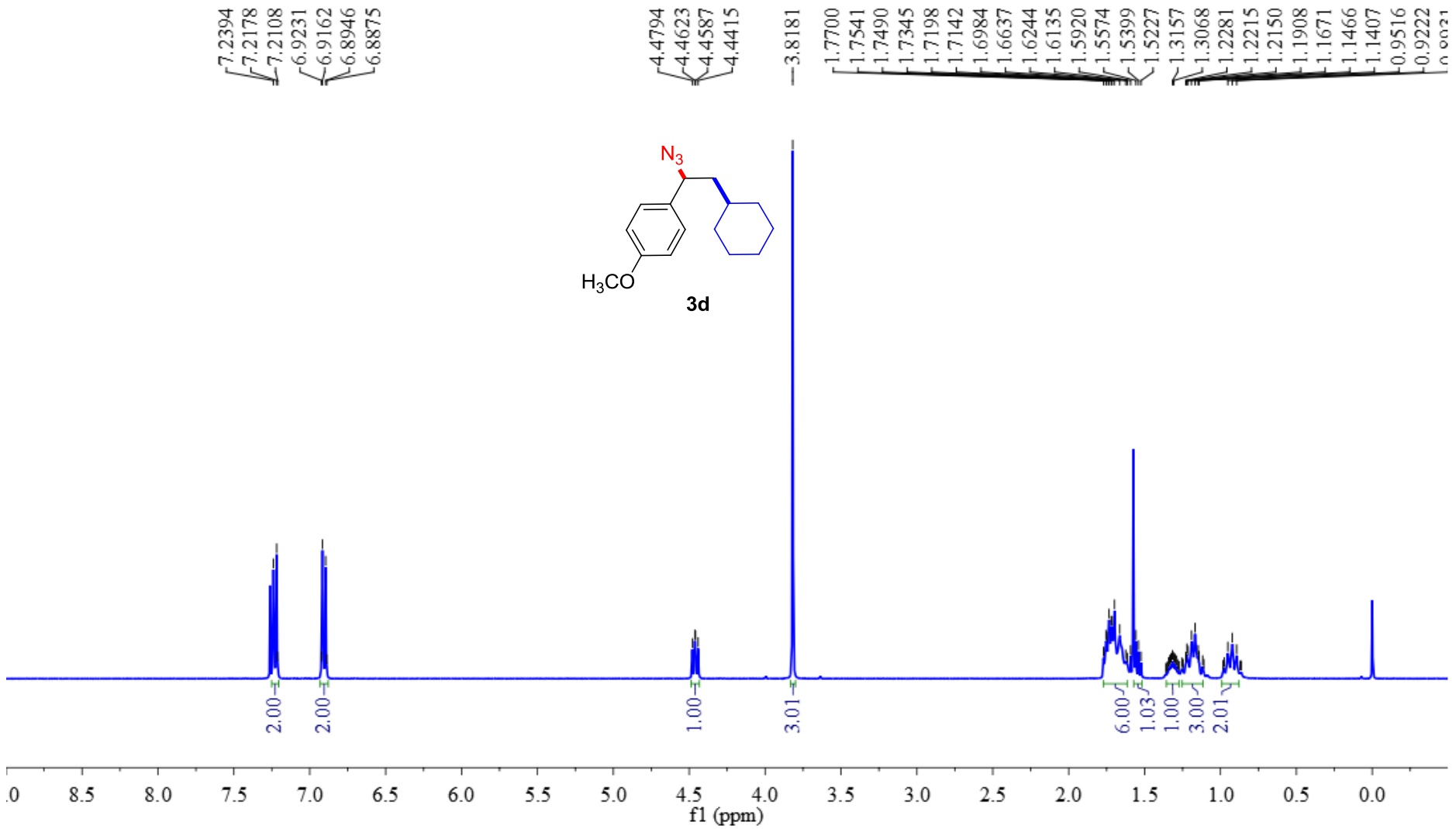
7.374
7.232
7.212
4.492
4.477
4.469
4.454
1.789
1.774
1.767
1.753
1.740
1.732
1.717
1.710
1.677
1.668
1.630
1.592
1.558
1.539
1.322
1.253
1.245
1.221
1.214
1.184
1.164
1.158
0.964
0.958
0.934
0.928
0.906
N3
t-Bu
3c
1.99
2.02
1.00
6.00
0.99
1.01
9.01
3.03
2.00
f1 (ppm)


151.168
137.303
126.640
125.780
63.626
43.772
34.708
34.479
33.648
32.902
31.450
26.625
26.270
26.170
N3
t-Bu
3c
f1 (ppm)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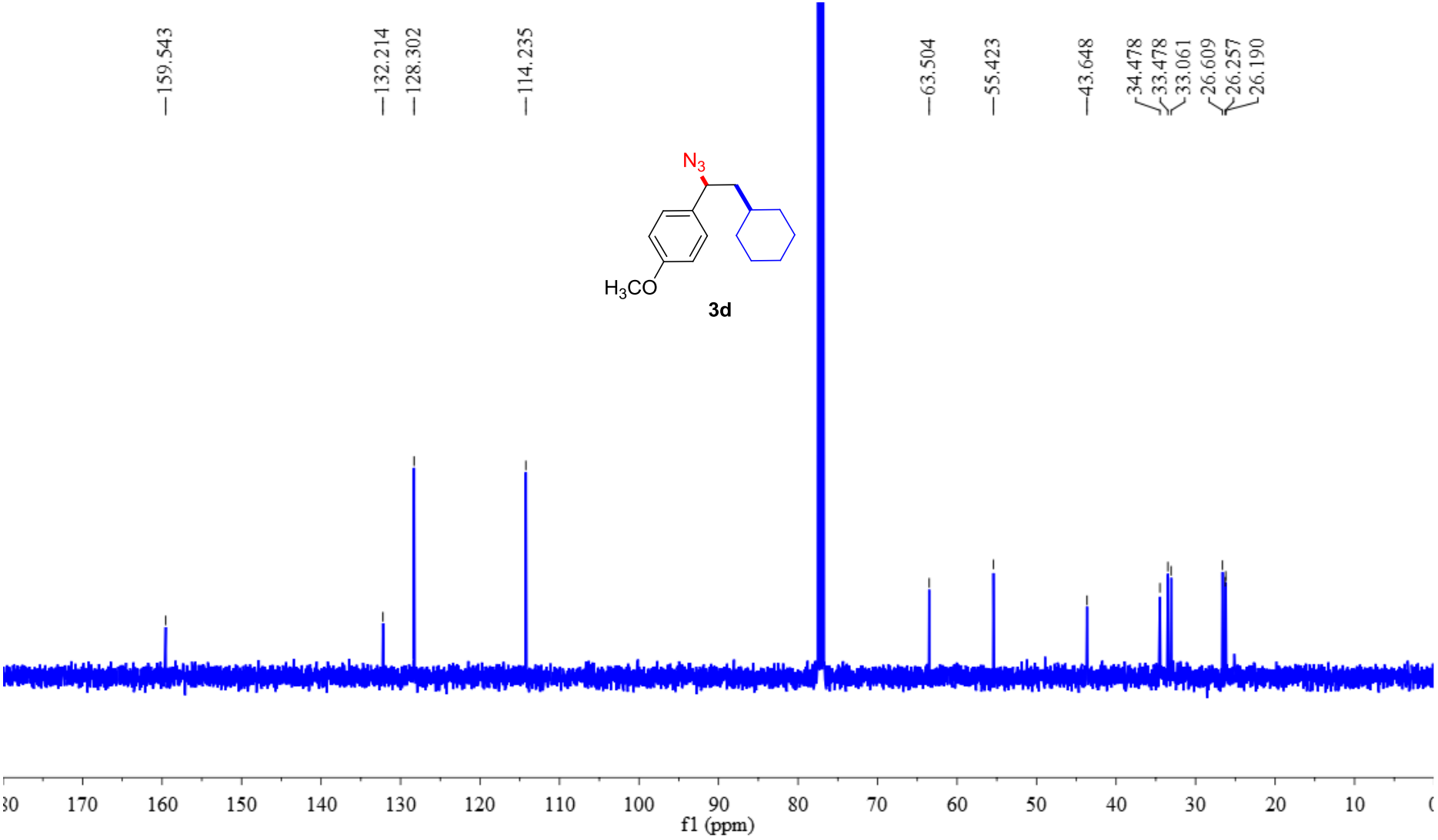
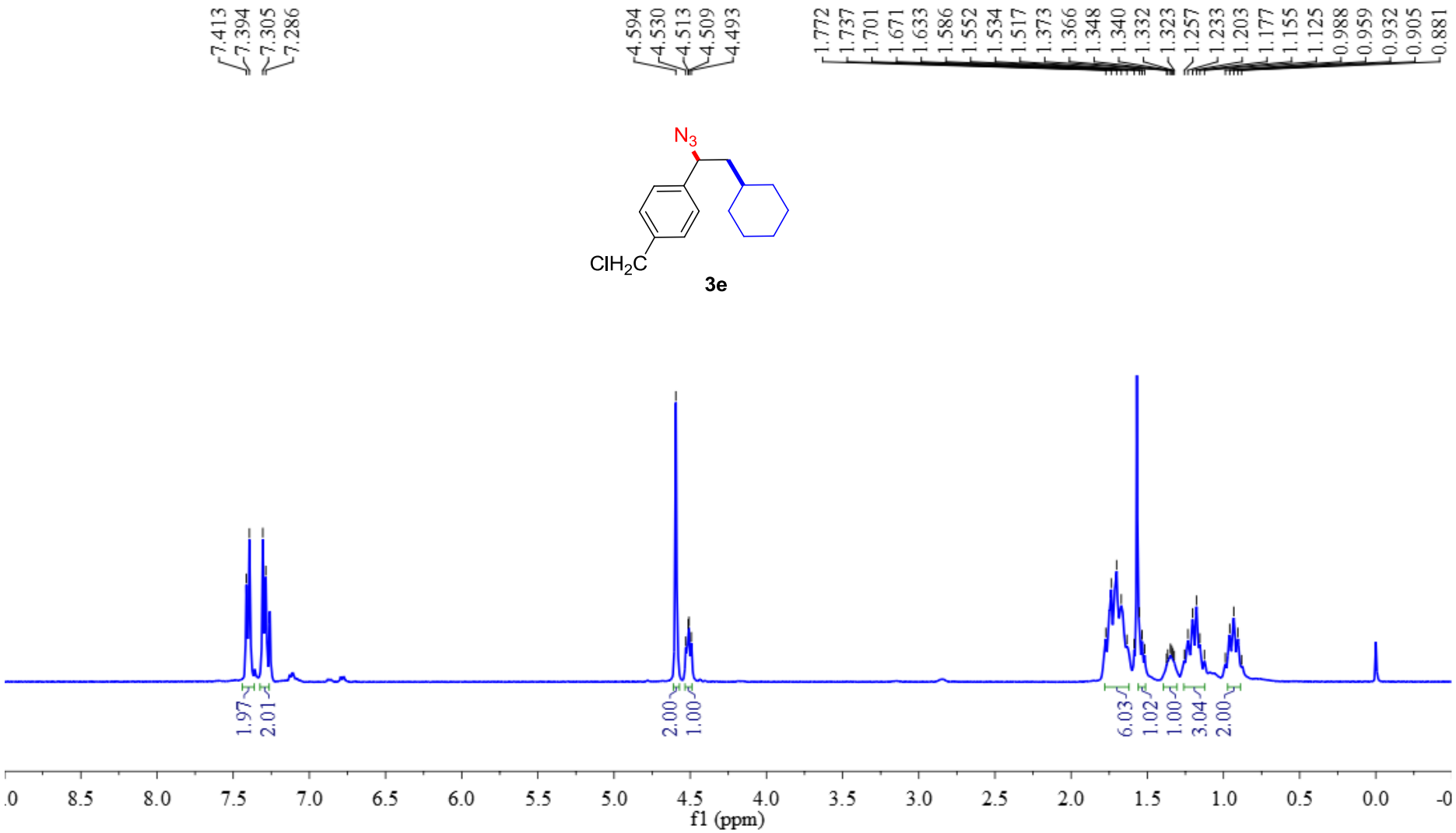
7.2178
7.2108
6.9231
6.9162
6.8946
6.8875
4.4794
4.4623
4.4587
4.4415
3.8181
1.7700
1.7541
1.7490
1.7345
1.7198
1.7142
1.6984
1.6637
1.6244
1.6135
1.5920
1.5574
1.5399
1.5227
1.3157
1.3068
1.2281
1.2215
1.2150
1.1908
1.1671
1.1466
1.1407
0.9516
0.9222
N3
H3CO
3d
2.00
2.00
1.00
3.01
6.00
1.03
1.00
3.00
2.01
f1 (ppm)


159.543
132.214
128.302
114.235
63.504
55.423
43.648
34.478
33.478
33.061
26.609
26.257
26.190
N3
H3CO
3d
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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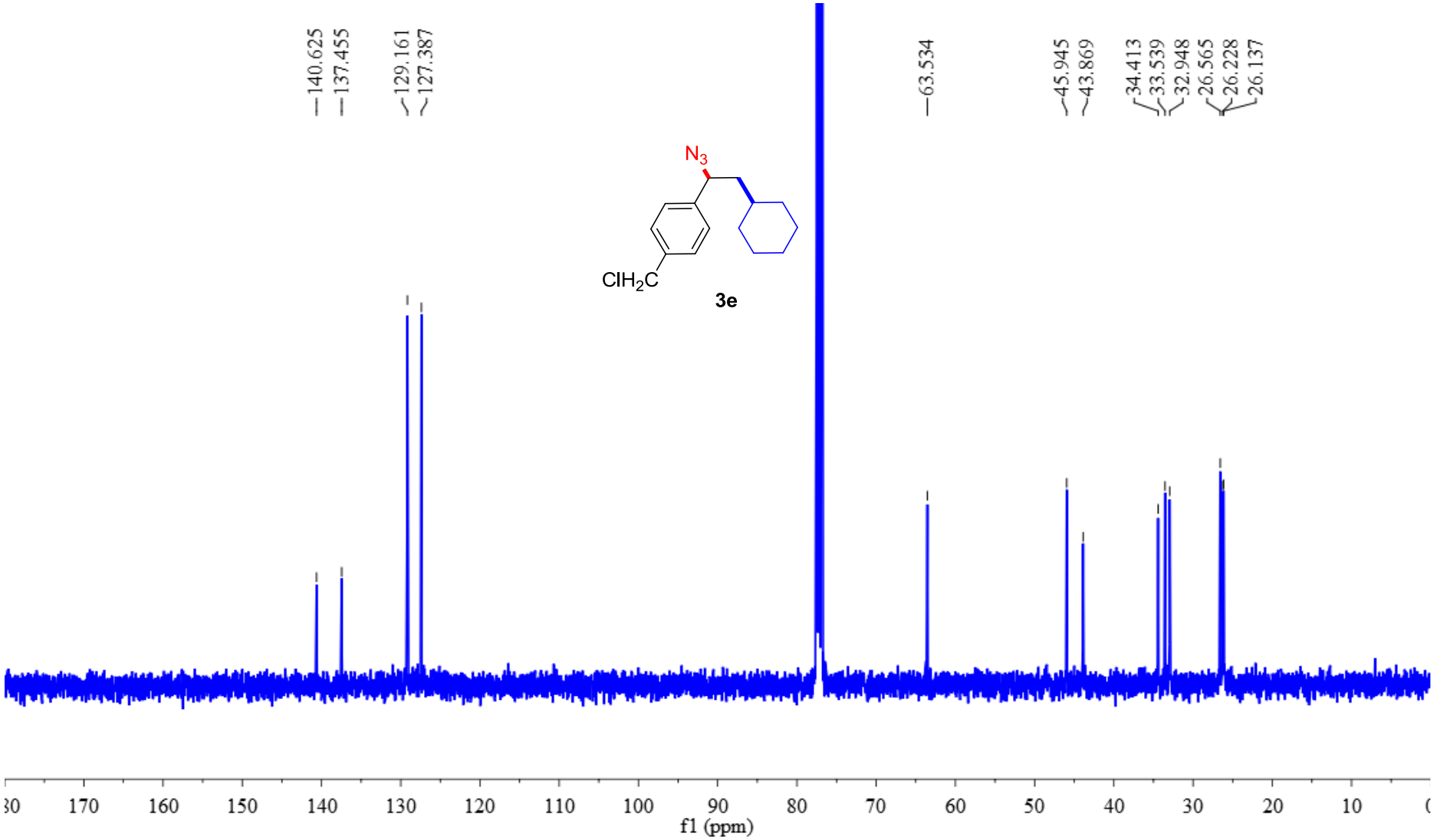
7.394
7.305
7.286
4.594
4.530
4.513
4.509
4.493
1.772
1.737
1.701
1.671
1.633
1.586
1.552
1.534
1.517
1.373
1.366
1.348
1.340
1.332
1.323
1.257
1.233
1.203
1.177
1.155
1.125
0.988
0.959
0.932
0.905
0.881
N3
ClH2C
1.97
2.01
2.00
1.00
6.03
1.02
1.00
3.04
2.00
f1 (ppm)

3e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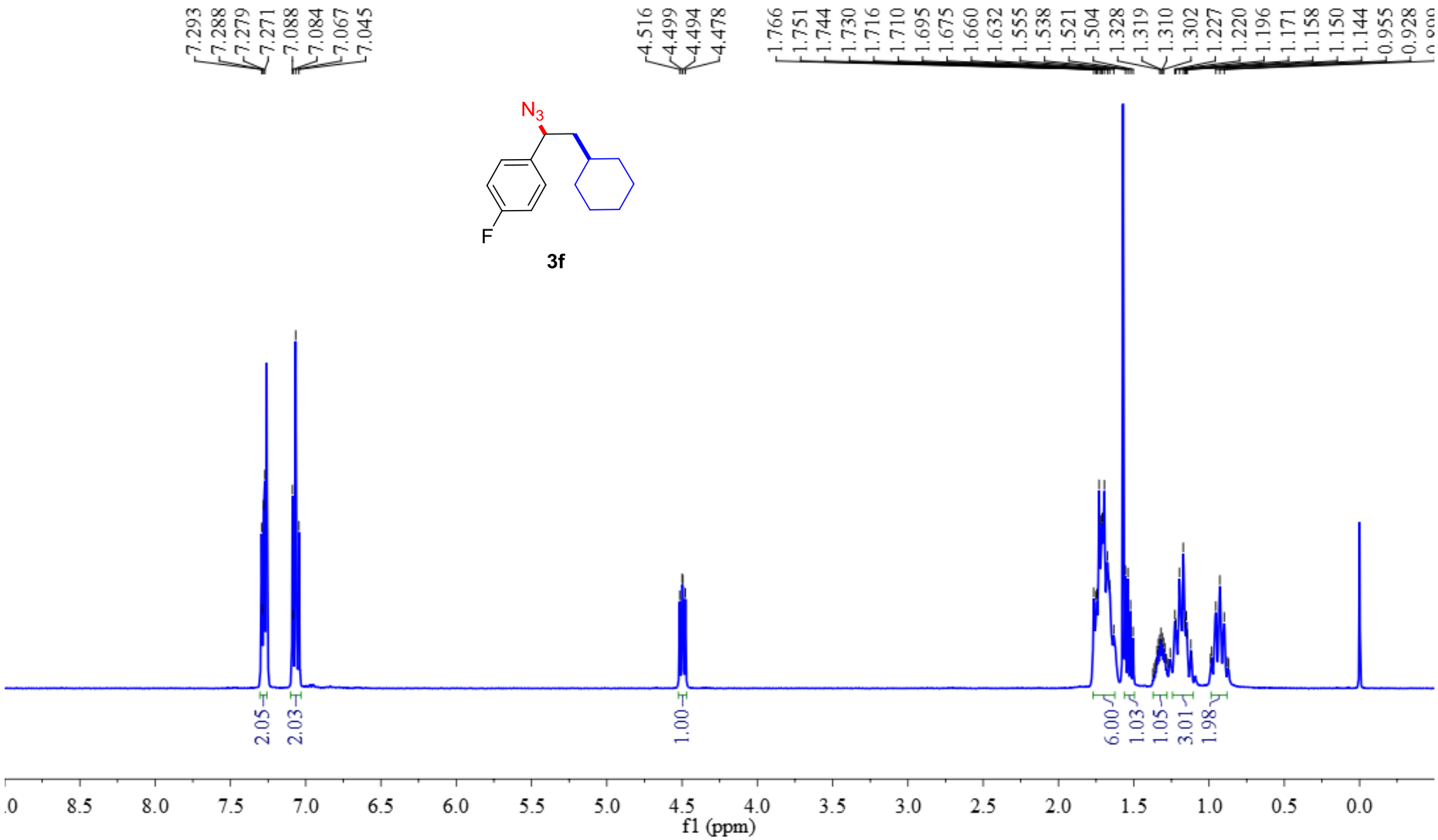
137.455
129.161
127.387
63.534
45.945
43.869
34.413
33.539
32.948
26.565
26.228
26.137
N3
ClH2C
3e
f1 (ppm)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7.279
7.271
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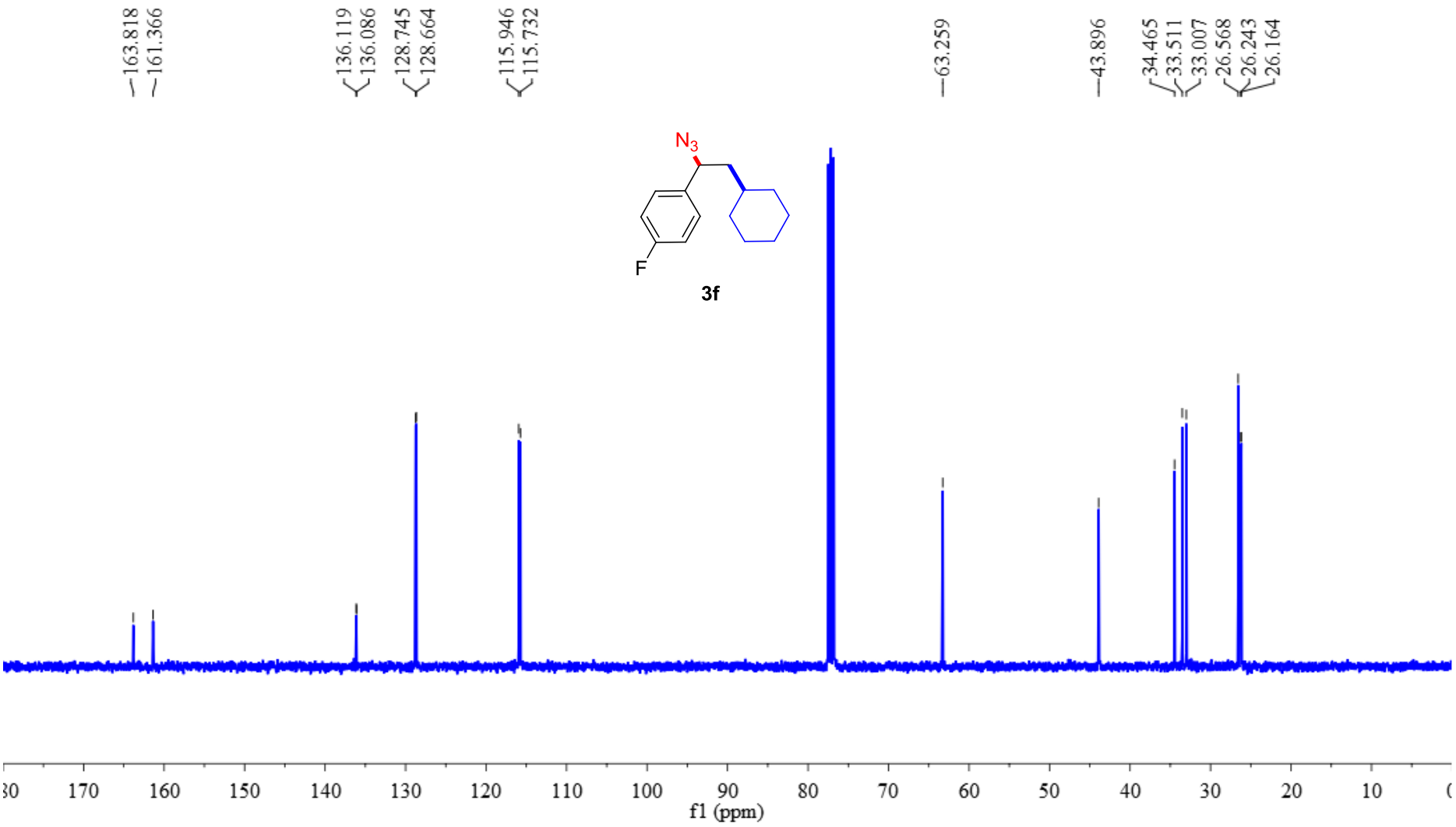
7.084
7.067
7.045
4.516
4.499
4.494
4.478
1.766
1.751
1.744
1.730
1.716
1.710
1.695
1.675
1.660
1.632
1.555
1.538
1.521
1.504
1.328
1.319
1.310
1.302
1.227
1.220
1.196
1.171
1.158
1.150
1.144
0.955
0.928
N3
F
3f
2.05
2.03
1.00
6.00
1.03
1.05
3.01
1.98
f1 (ppm)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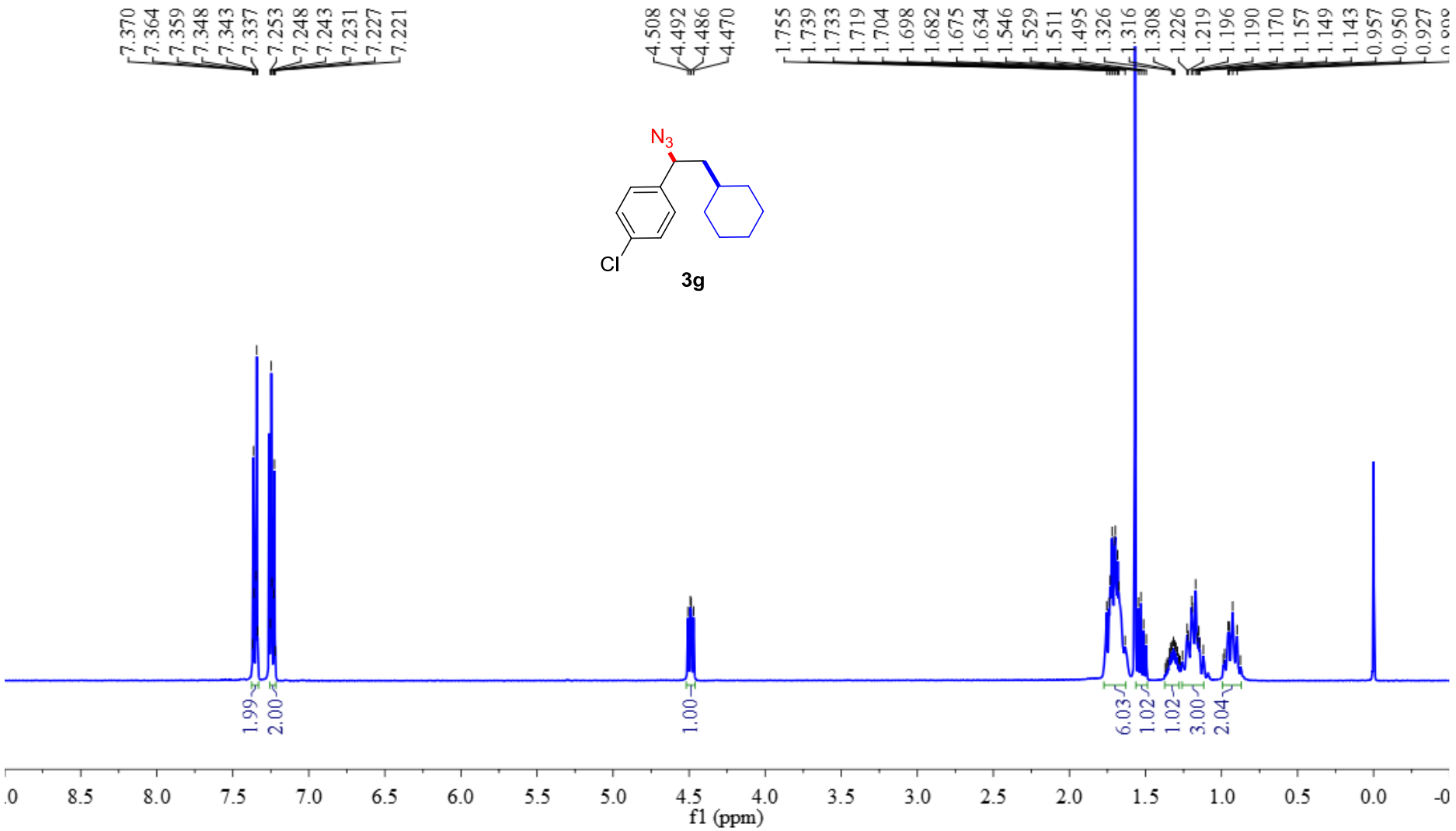
161.366
136.119
136.086
128.745
128.664
115.946
115.732
63.259
43.896
34.465
33.511
33.007
26.568
26.243
26.164
N3
F
3f
f1 (ppm)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7.337
7.253
7.248
7.243
7.231
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7.221
4.508
4.492
4.486
4.470
1.755
1.739
1.733
1.719
1.704
1.698
1.682
1.675
1.634
1.546
1.529
1.511
1.495
1.326
1.316
1.308
1.226
1.219
1.196
1.190
1.170
1.157
1.149
1.143
0.957
0.950
0.927
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1.00
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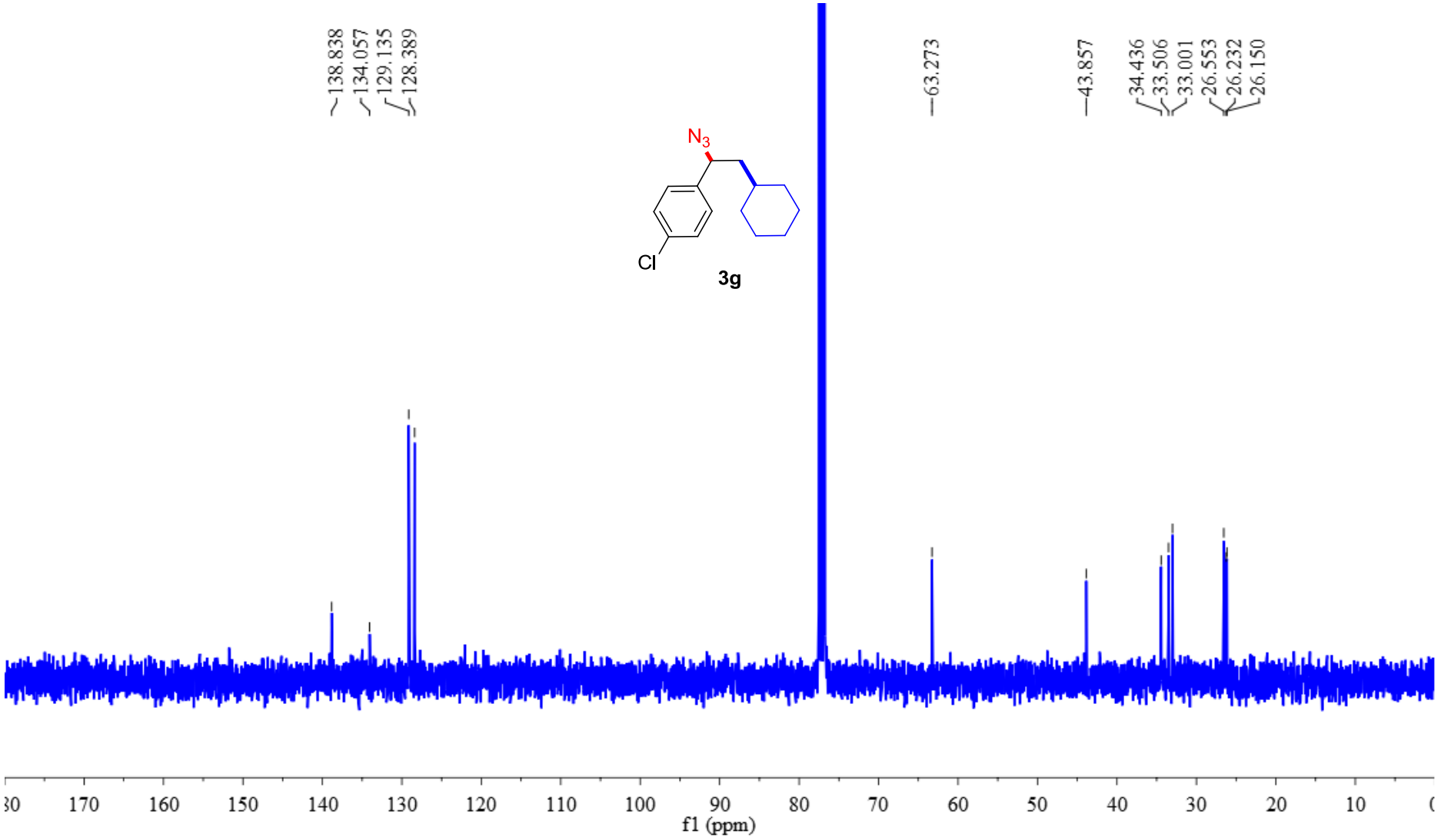
1.02
1.02
3.00
2.04
f1 (ppm)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128.389
63.273
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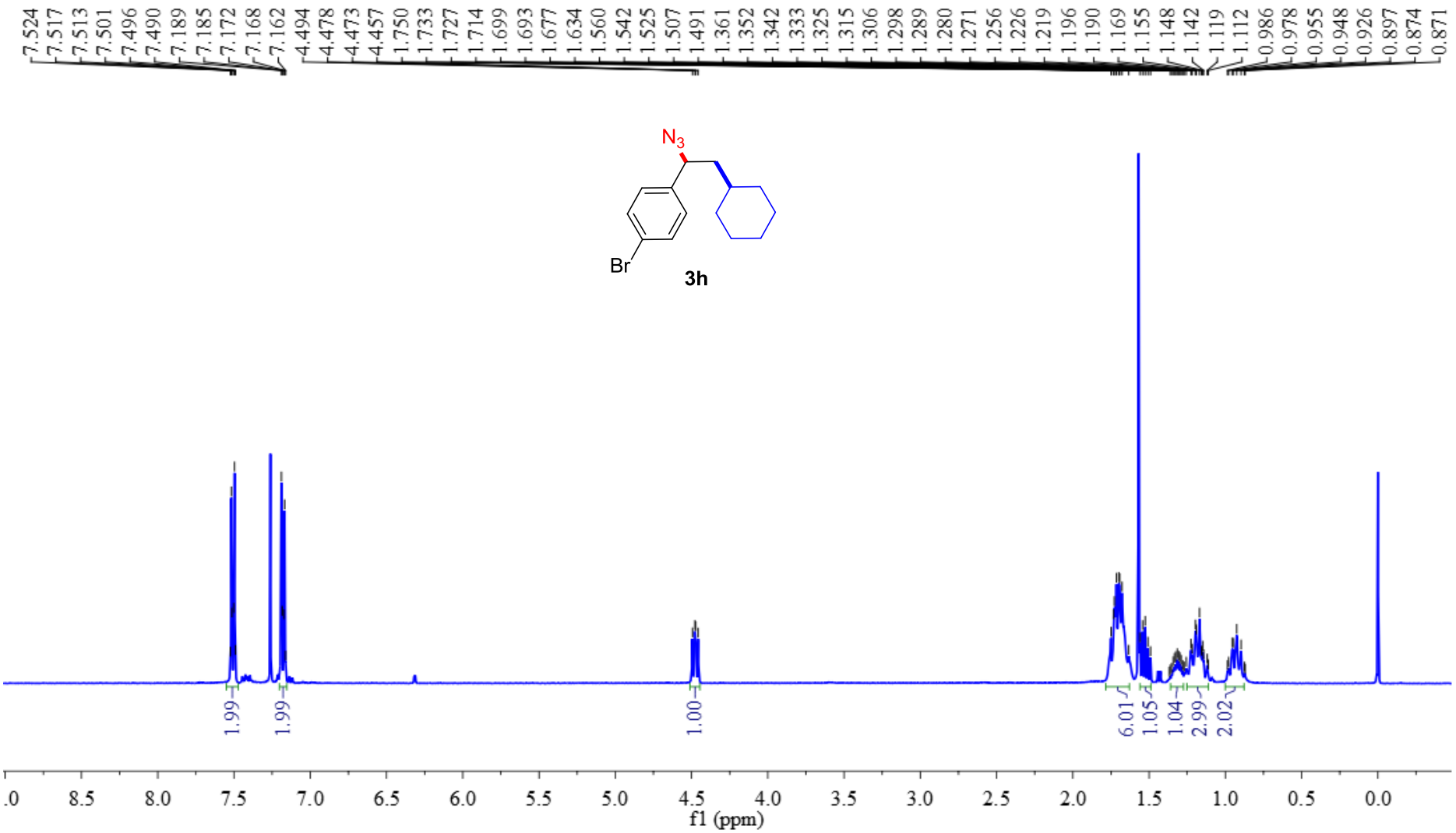
33.506
33.001
26.553
26.232
26.150
N3
Cl
3g
f1 (ppm)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7.513
7.501
7.496
7.490
7.189
7.185
7.172
7.168
7.162
4.494
4.478
4.473
4.457
1.750
1.733
1.727
1.714
1.699
1.693
1.677
1.634
1.560
1.542
1.525
1.507
1.491
1.361
1.352
1.342
1.333
1.325
1.315
1.306
1.298
1.289
1.280
1.271
1.256
1.226
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1.196
1.190
1.169
1.155
1.148
1.142
1.119
1.112
0.986
0.978
0.955
0.948
0.926
0.897
0.874
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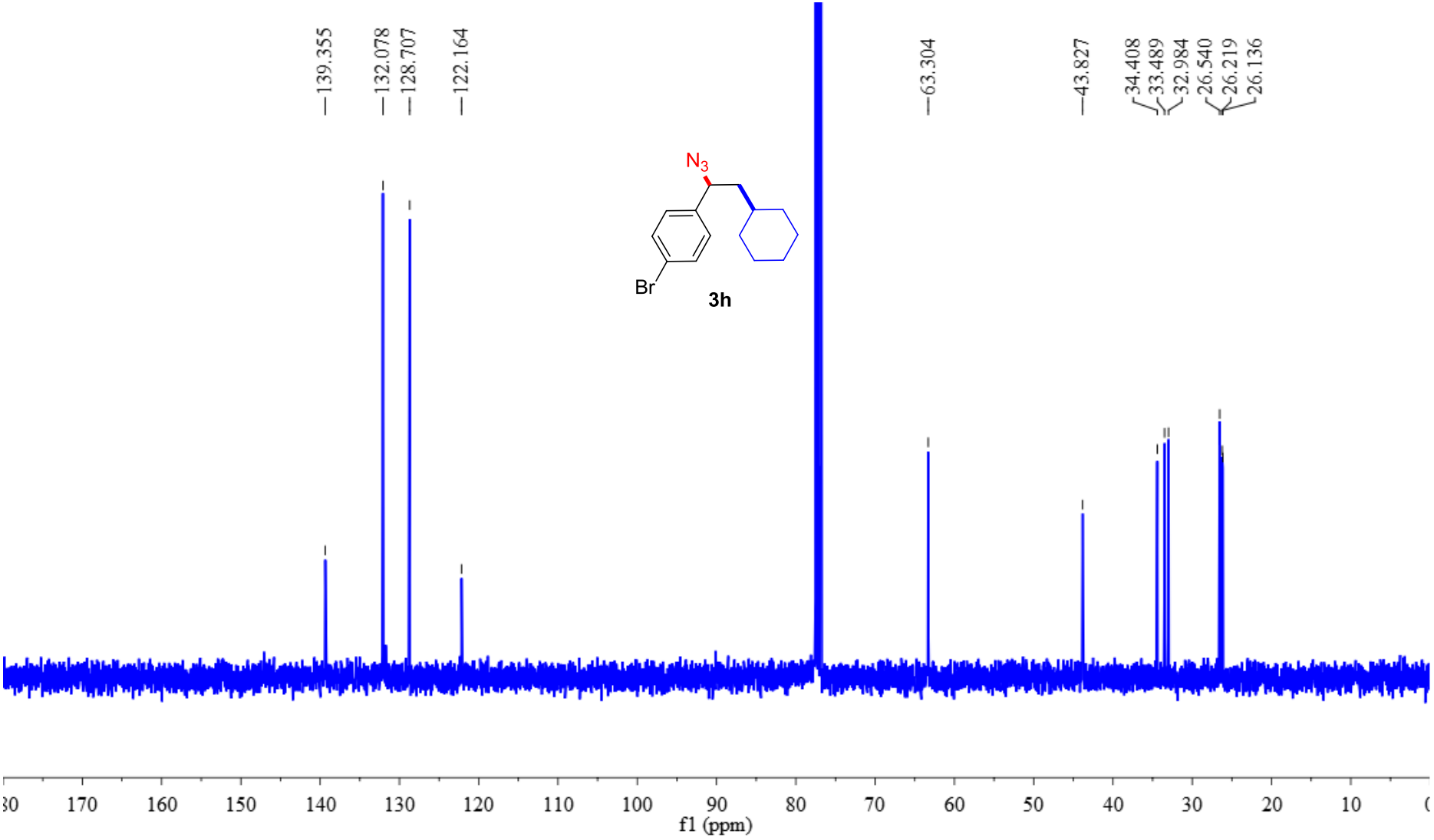
1.99
1.00
6.01
1.05
1.04
2.99
2.02
f1 (ppm)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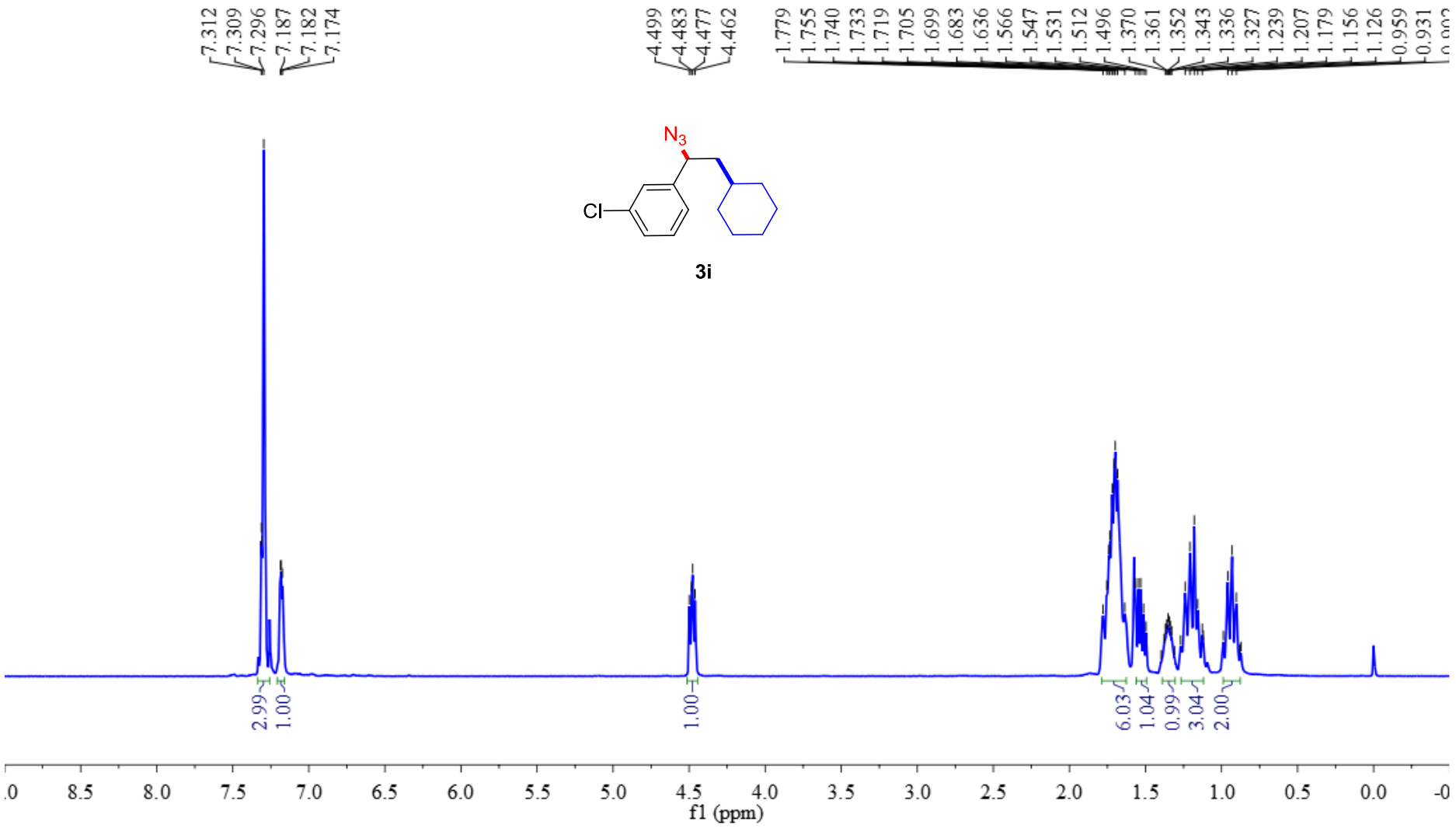
26.219
26.136
N3
Br
3h
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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4.483
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1.740
1.733
1.719
1.705
1.699
1.683
1.636
1.566
1.547
1.531
1.512
1.496
1.370
1.361
1.352
1.343
1.336
1.327
1.239
1.207
1.179
1.156
1.126
0.959
0.931
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Cl
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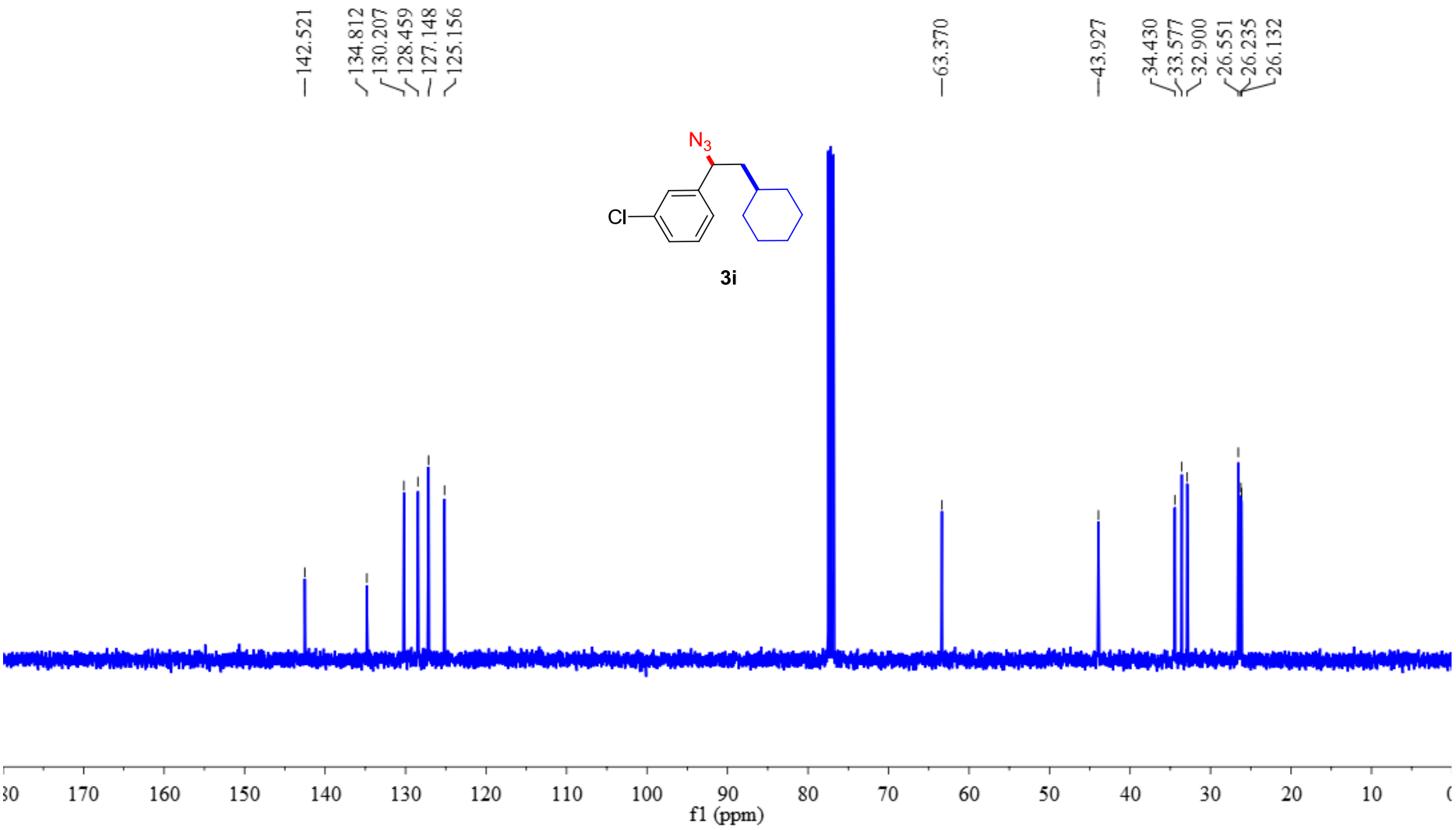
3.04
2.00
f1 (ppm)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63.370
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33.577
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26.132
N3
Cl
3i
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140
130
120
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100
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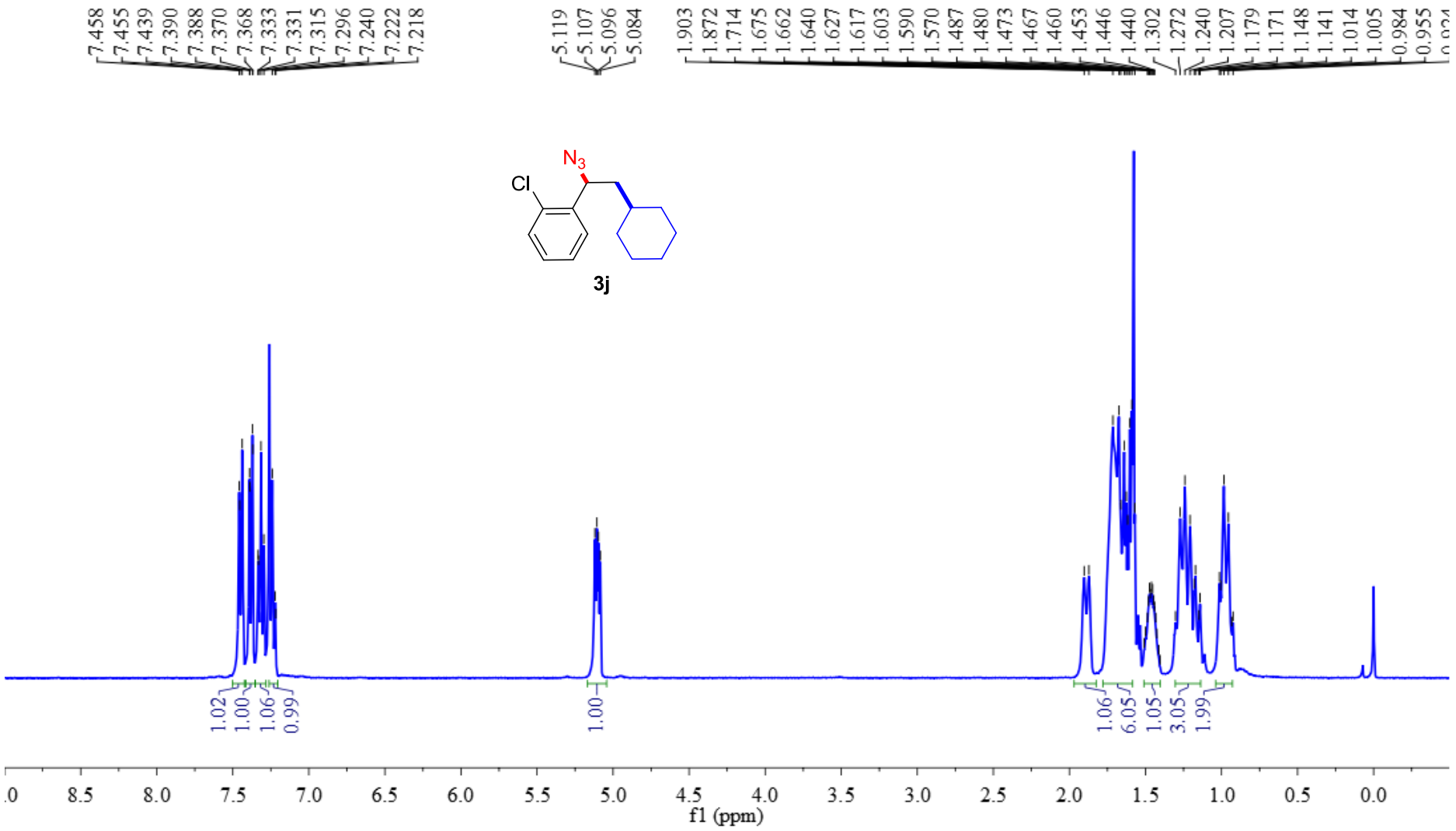
80
70
60
50
40
30
20
10
f1 (ppm)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7.296
7.240
7.222
7.218
5.119
5.107
5.096
5.084
1.903
1.872
1.714
1.675
1.662
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1.603
1.590
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1.453
1.446
1.440
1.302
1.272
1.240
1.207
1.179
1.171
1.148
1.141
1.014
1.005
0.984
0.955
N3
Cl
3j
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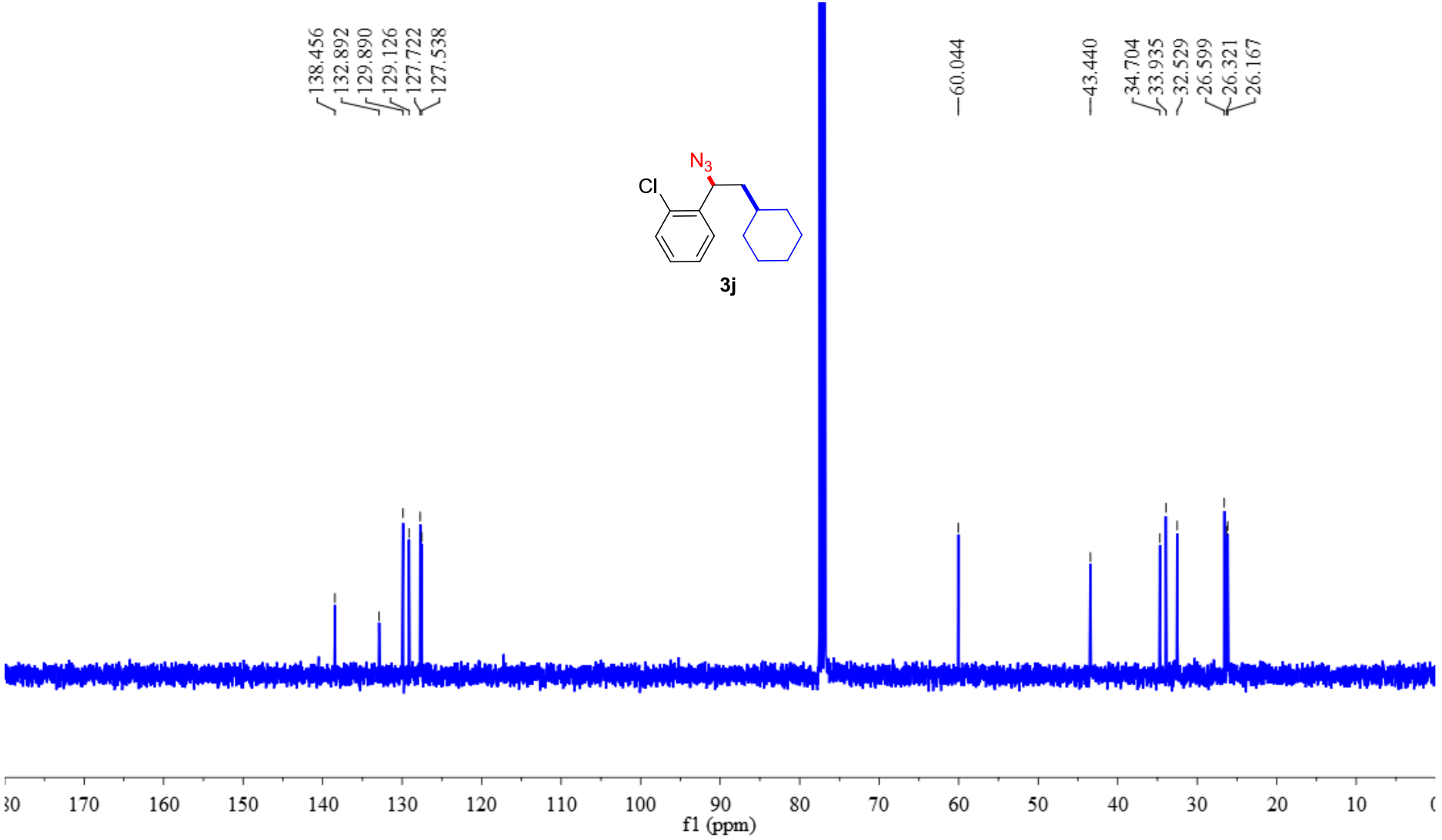
1.06
0.99
1.00
1.06
6.05
1.05
3.05
1.99
f1 (ppm)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129.126
127.722
127.538
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33.935
32.529
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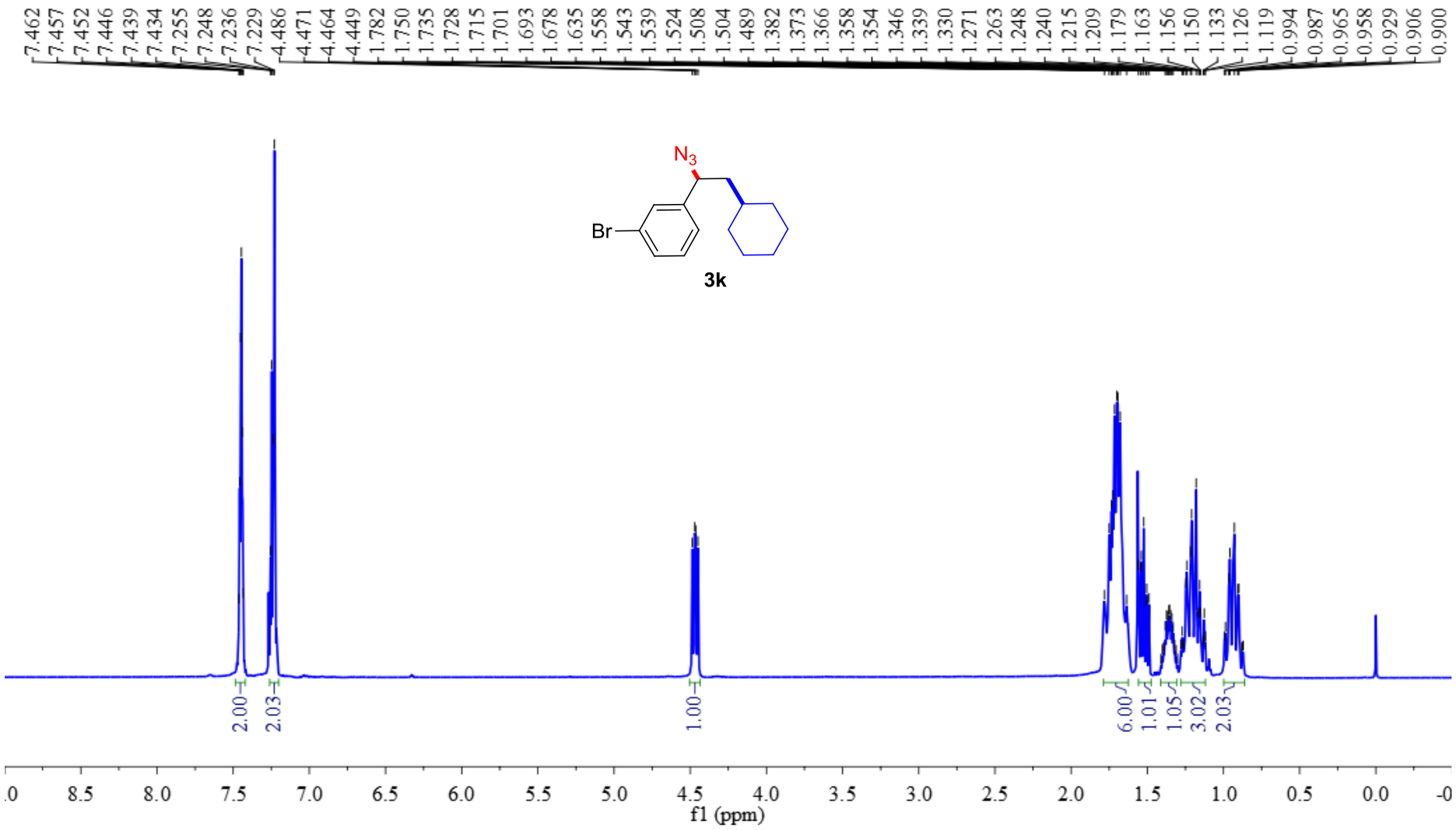
26.321
26.167
N3
Cl
3j
f1 (ppm)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7.229
4.486
4.471
4.464
4.449
1.782
1.750
1.735
1.728
1.715
1.701
1.693
1.678
1.635
1.558
1.543
1.539
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1.508
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1.358
1.354
1.346
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1.271
1.263
1.248
1.240
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1.209
1.179
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1.156
1.150
1.133
1.126
1.119
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0.987
0.965
0.958
0.929
0.906
0.900
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Br
3k
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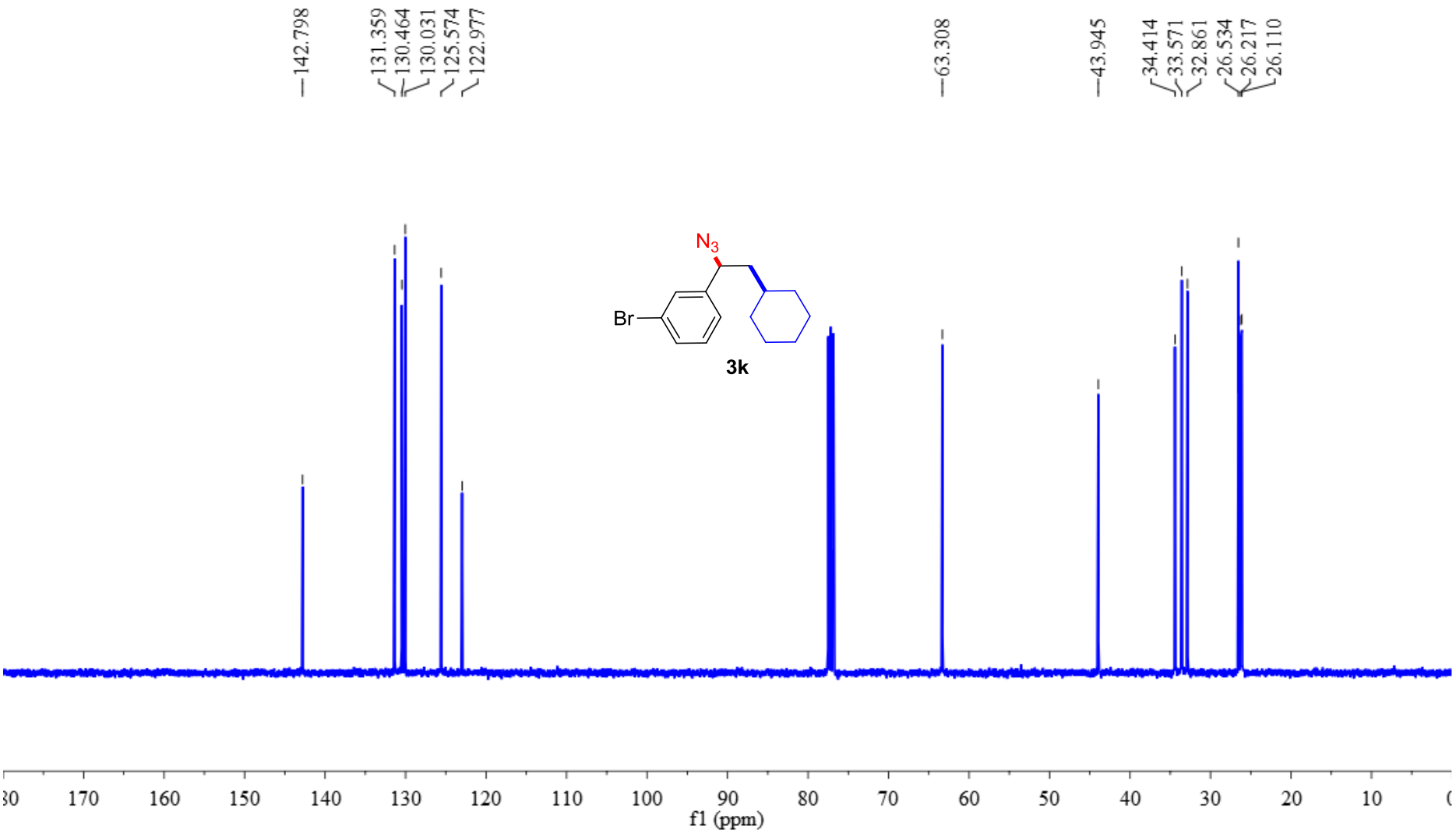
2.03
1.00
6.00
1.01
1.05
3.02
2.03
f1 (ppm)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130.031
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122.977
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43.945
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33.571
32.861
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26.110
N3
Br
3k
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150
140
130
120
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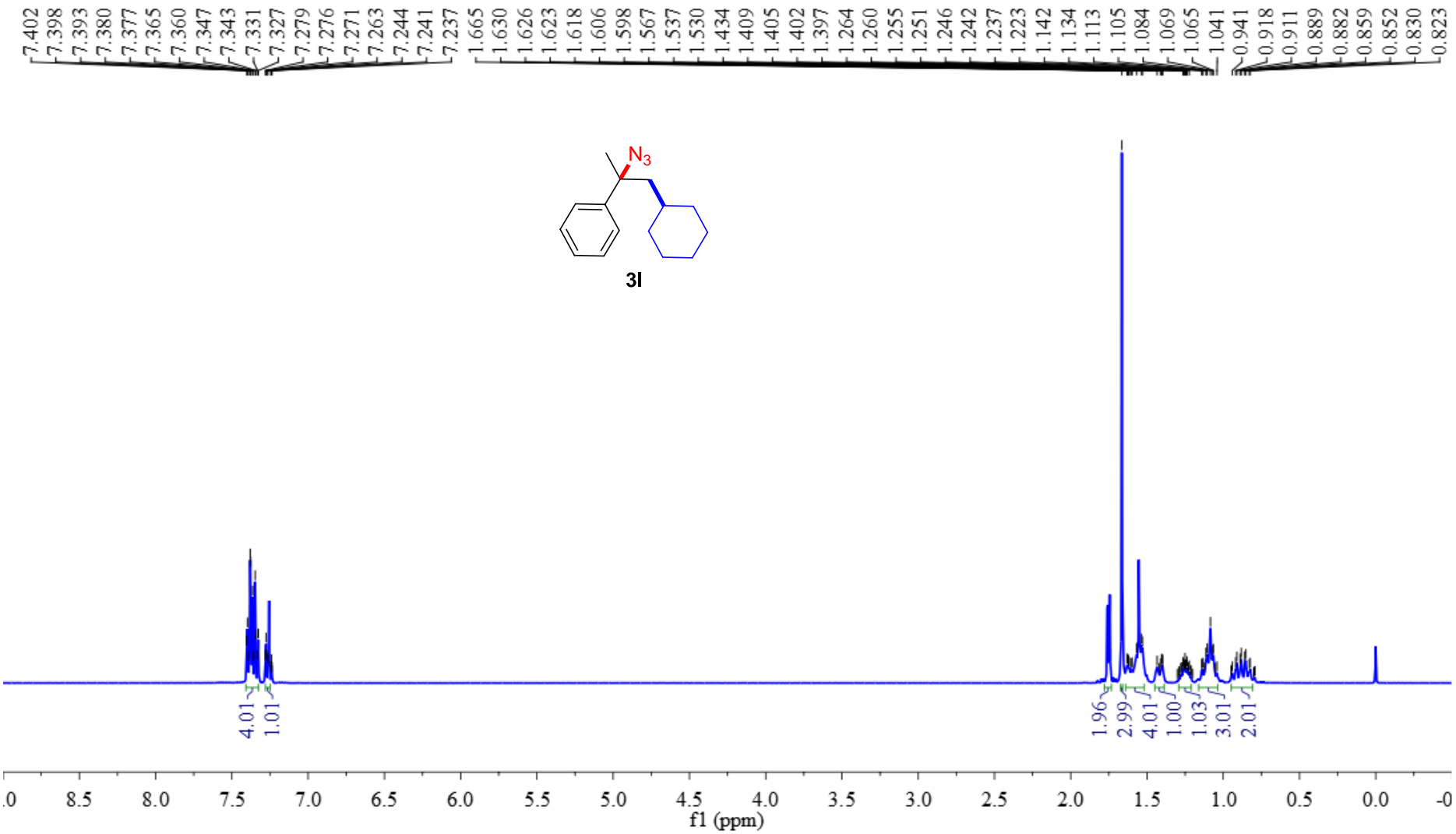
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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7.377
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7.347
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7.327
7.279
7.276
7.271
7.263
7.244
7.241
7.237
1.665
1.630
1.626
1.623
1.618
1.606
1.598
1.567
1.537
1.530
1.434
1.409
1.405
1.402
1.397
1.264
1.260
1.255
1.251
1.246
1.242
1.237
1.223
1.142
1.134
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1.069
1.065
1.041
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0.889
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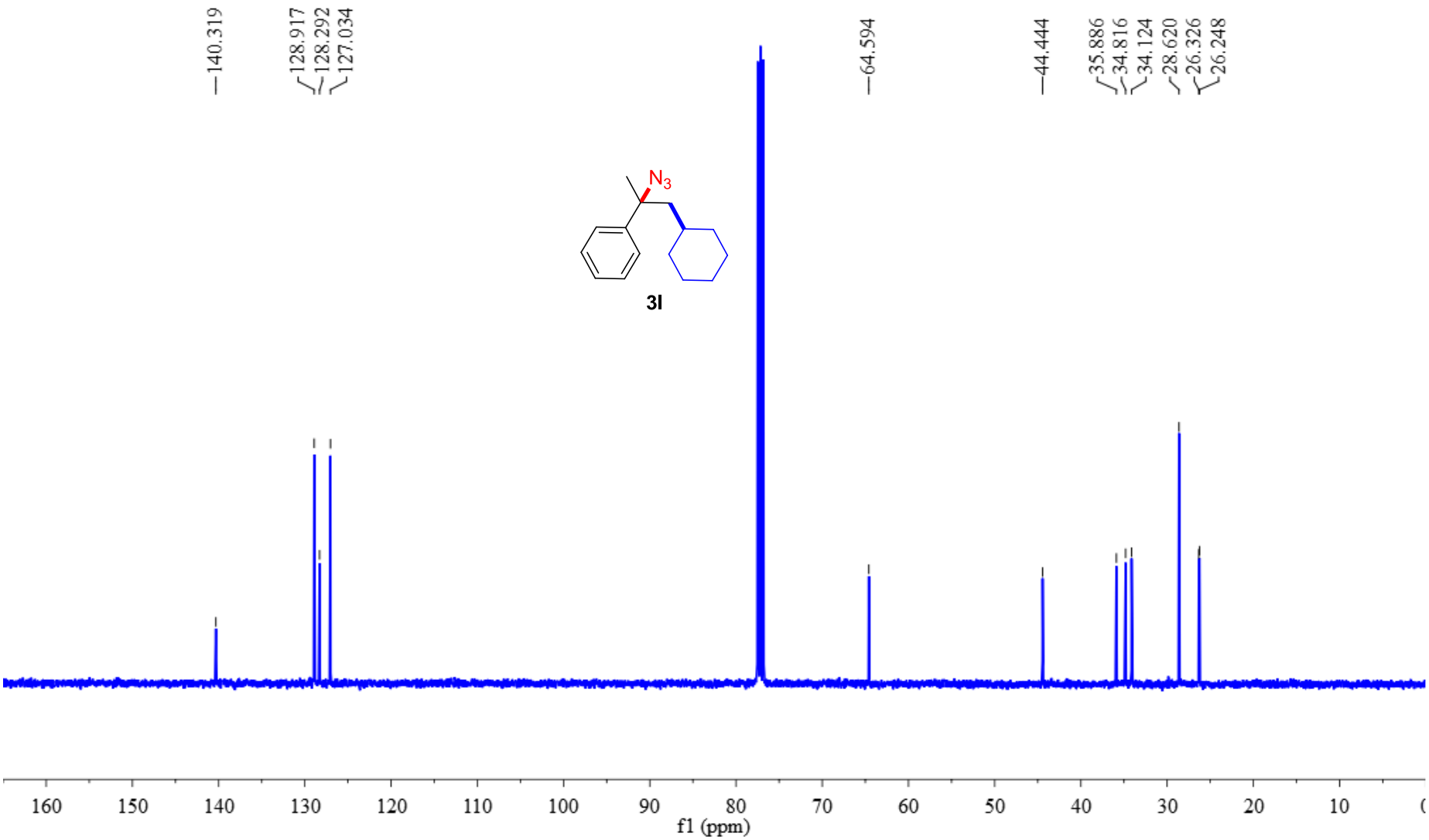
0.852
0.830
0.823
N3
3l
4.01
1.01
1.96
2.99
4.01
1.00
1.03
3.01
2.01
f1 (ppm)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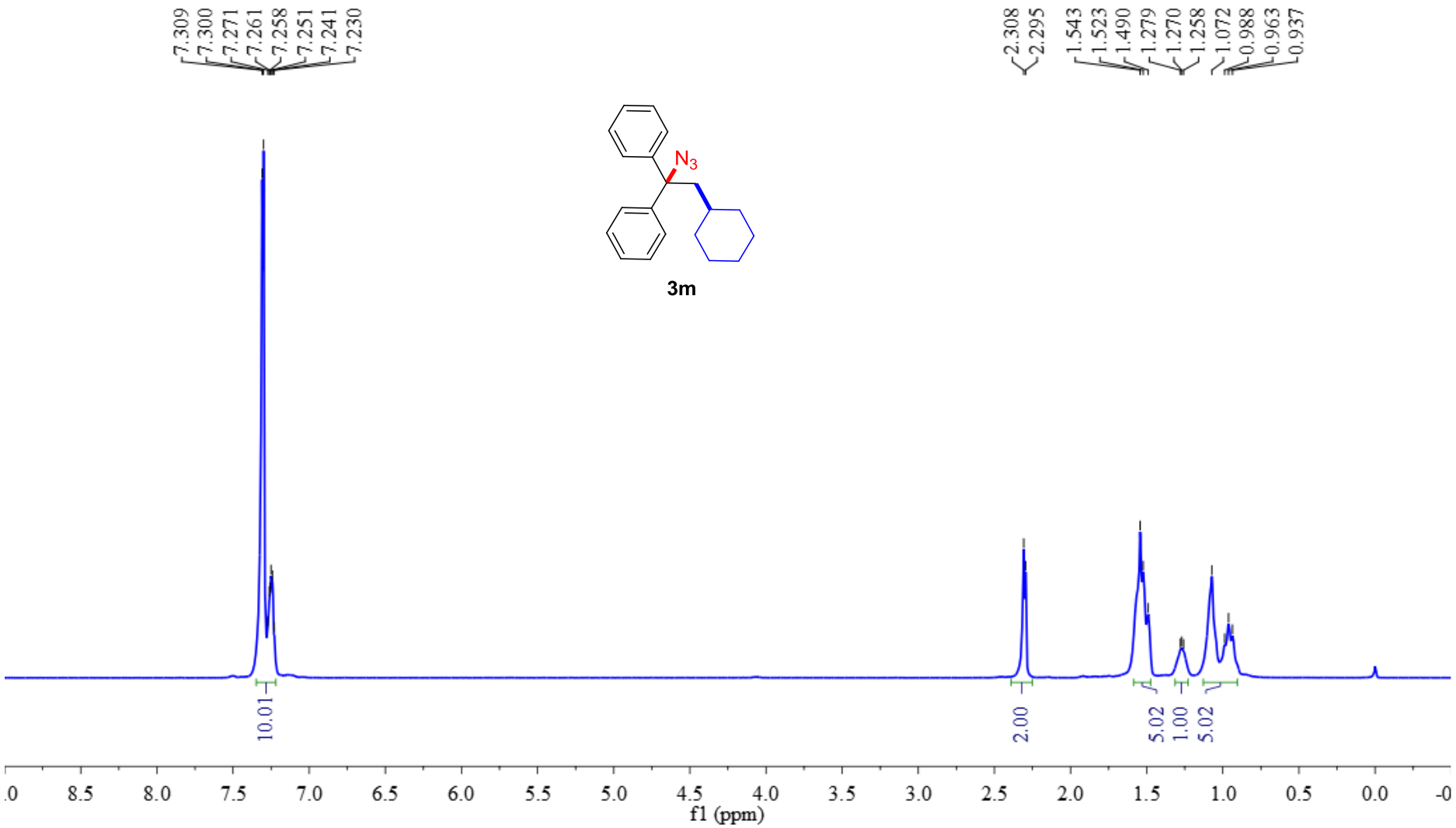
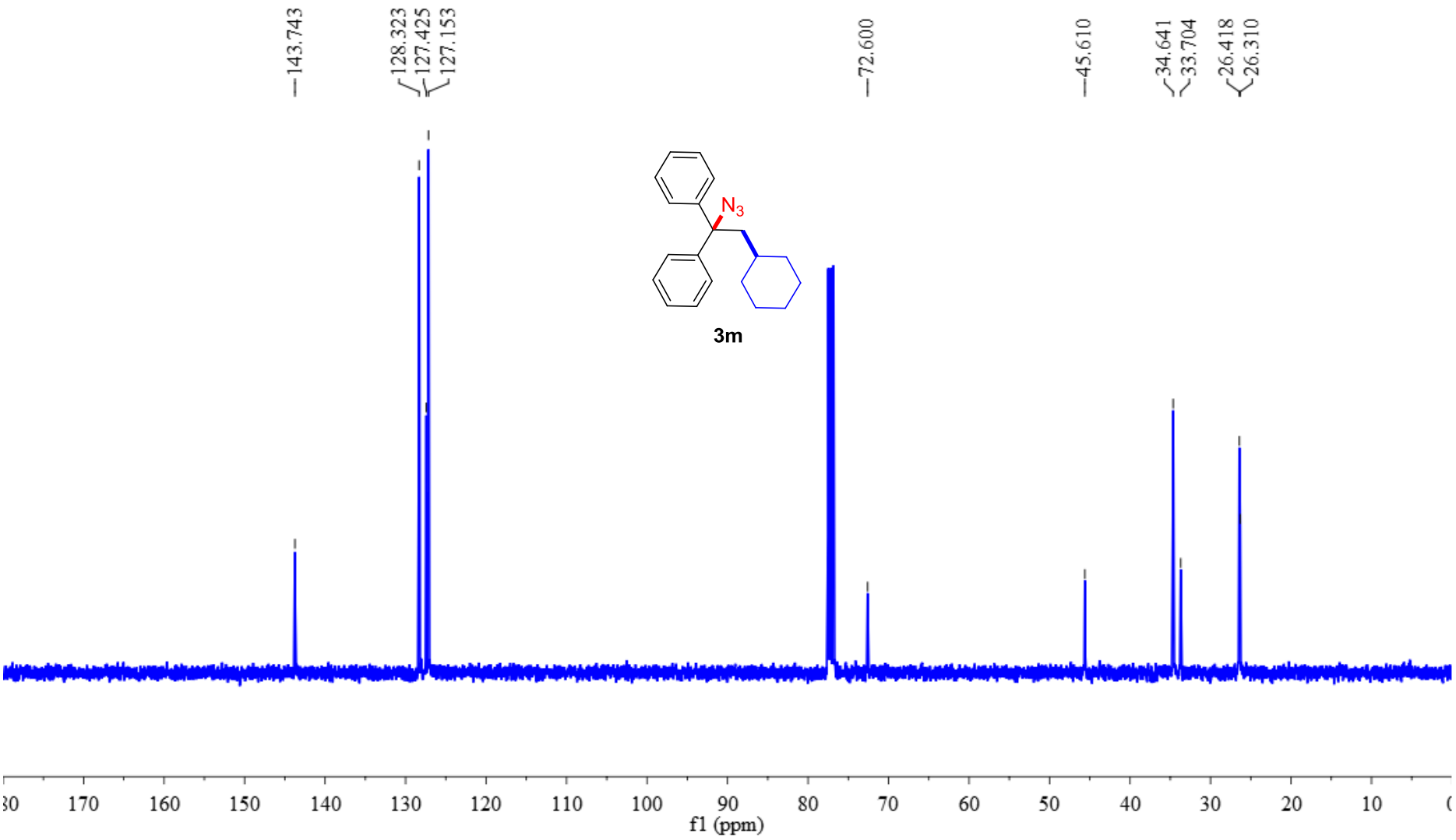
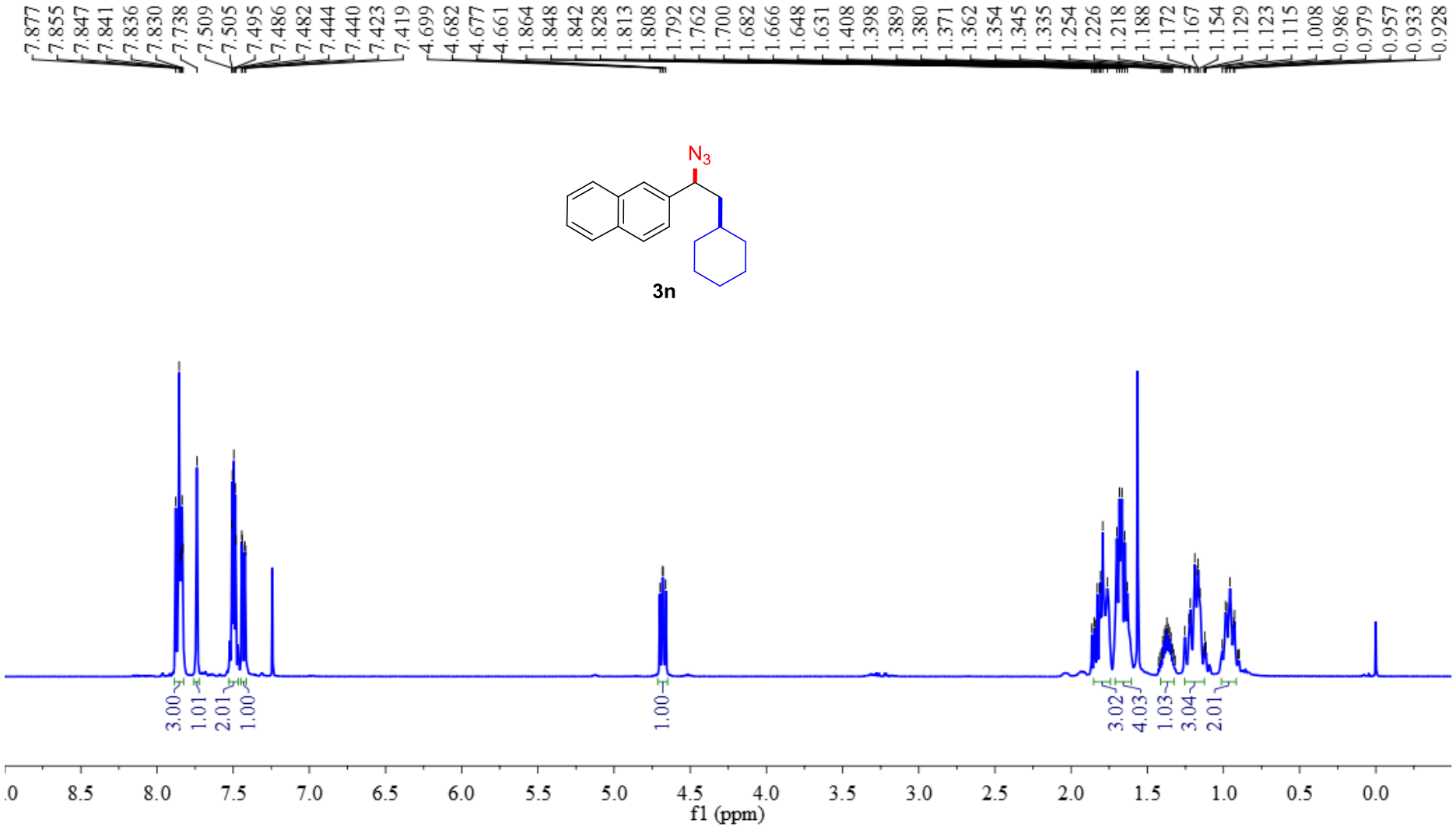
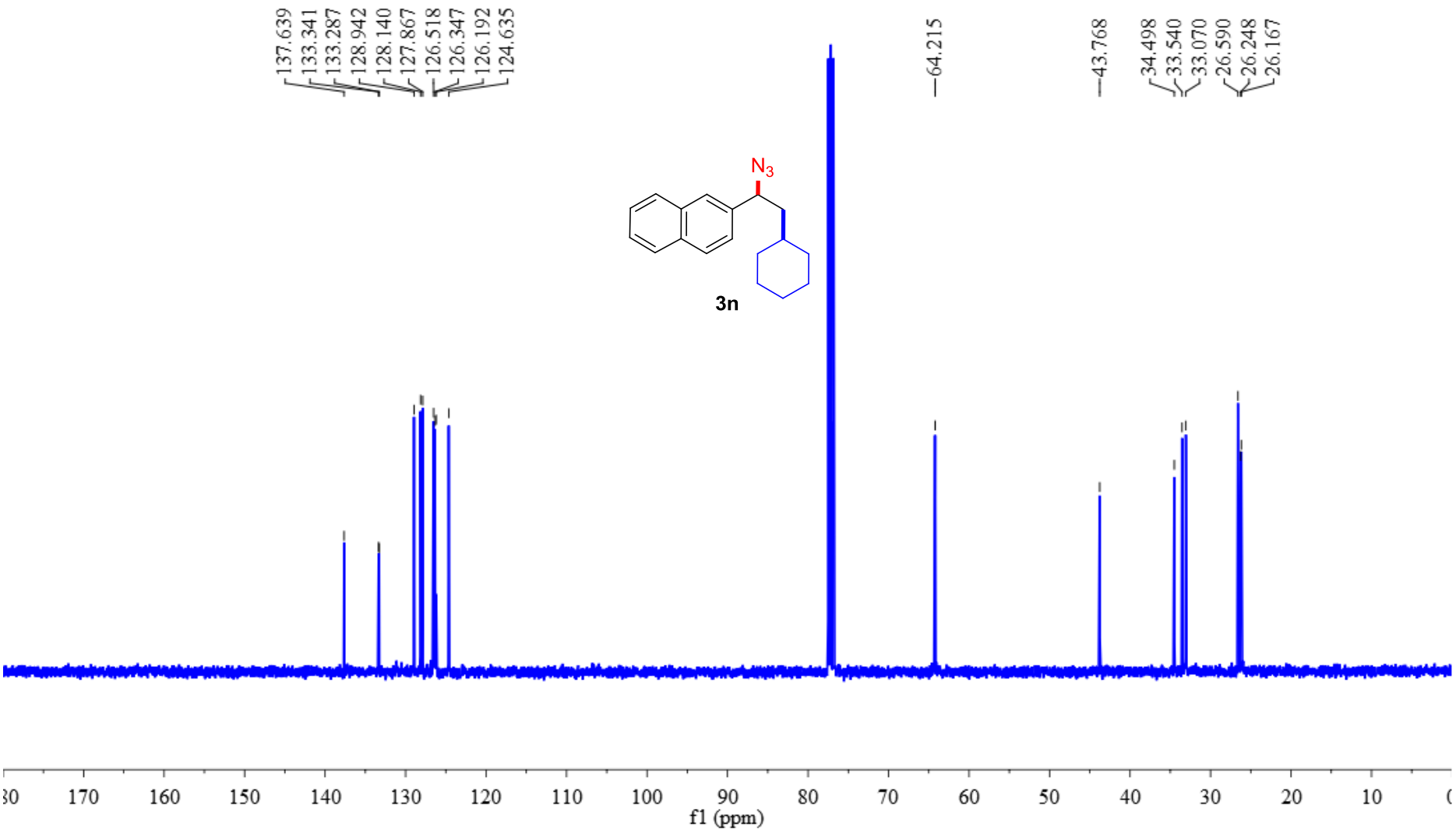
128.917
128.292
127.034
64.594
44.444
35.886
34.816
34.124
28.620
26.326
26.248
N3
3l
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


7.309
7.300
7.271
7.261
7.258
7.251
7.241
7.230
2.308
2.295
1.543
1.523
1.490
1.279
1.270
1.258
1.072
0.988
0.963
0.937
N3
10.01
2.00
5.02
1.00
5.02
f1 (ppm)

3m


143.743
128.323
127.425
127.153
72.600
45.610
34.641
33.704
26.418
26.310
N3
3m
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


7.877
7.855
7.847
7.841
7.836
7.830
7.738
7.509
7.505
7.495
7.486
7.482
7.444
7.440
7.423
7.419
4.699
4.682
4.677
4.661
1.864
1.848
1.842
1.828
1.813
1.808
1.792
1.762
1.700
1.682
1.666
1.648
1.631
1.408
1.398
1.389
1.380
1.371
1.362
1.354
1.345
1.335
1.254
1.226
1.218
1.188
1.172
1.167
1.154
1.129
1.123
1.115
1.008
0.986
0.979
0.957
0.933
0.928
N3
3n
3.00
1.01
2.01
1.00
1.00
3.02
4.03
1.03
3.04
2.01
f1 (ppm)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133.341
133.287
128.942
128.140
127.867
126.518
126.347
126.192
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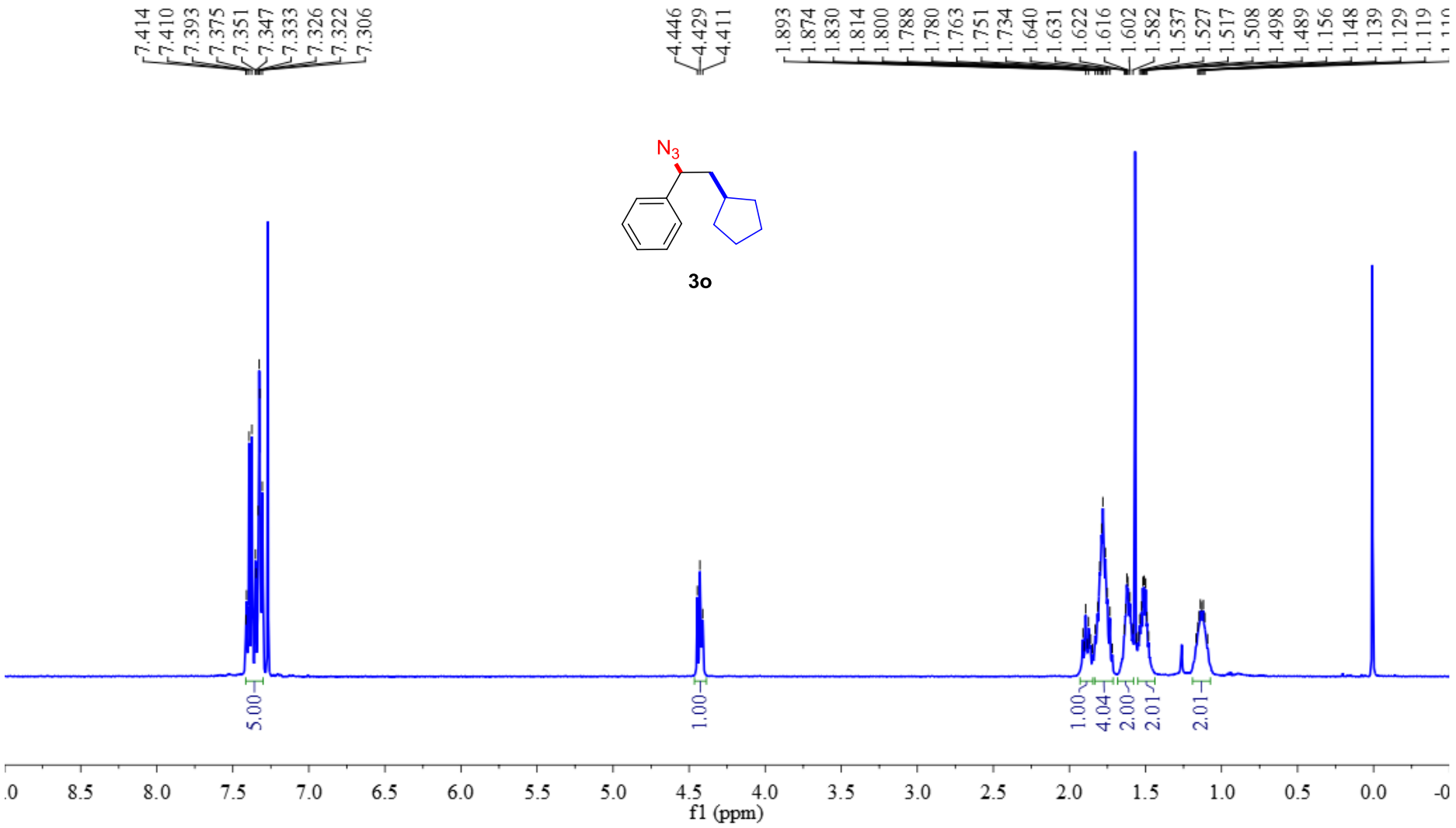
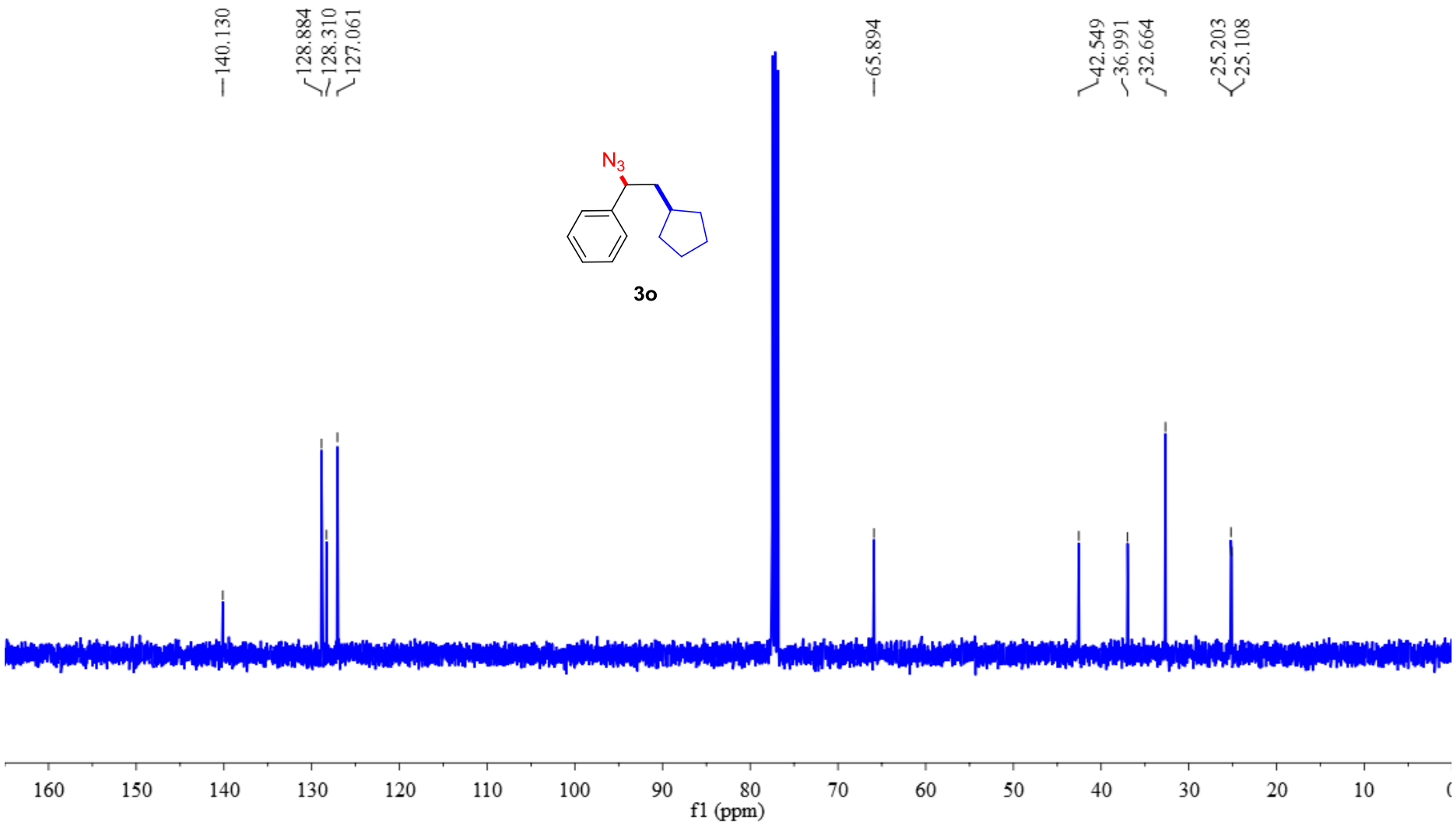
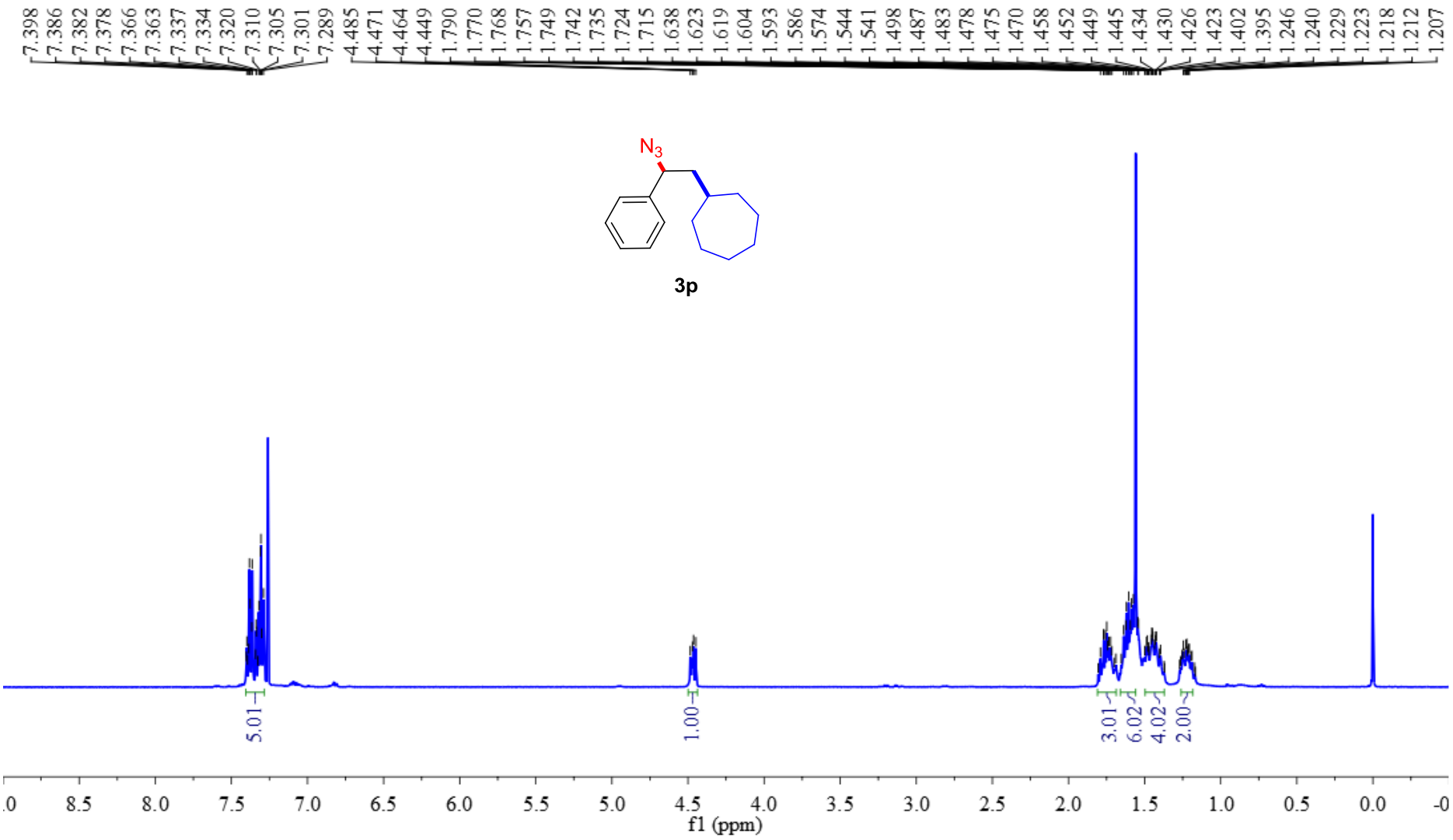
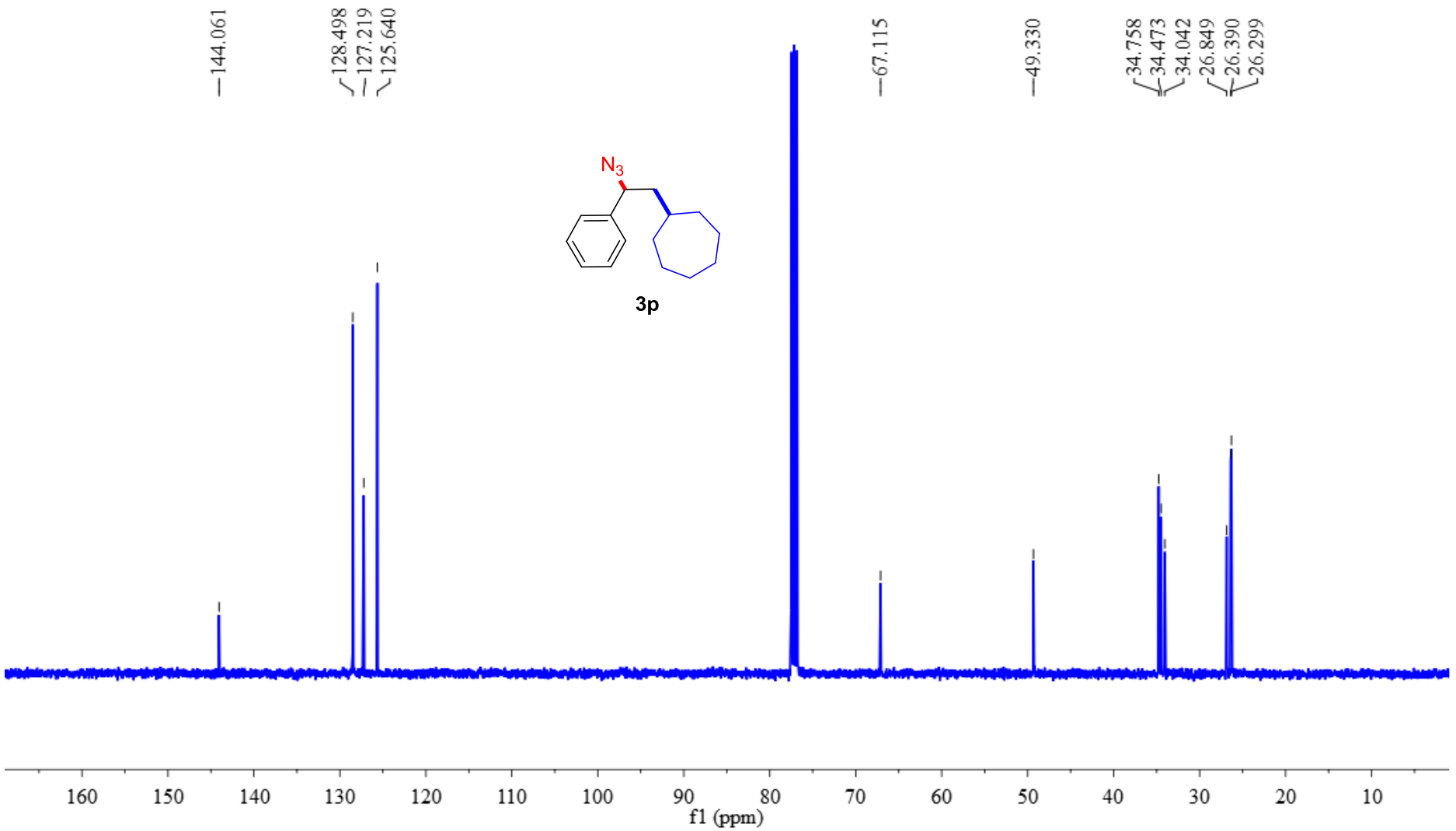
64.215
43.768
34.498
33.540
33.070
26.590
26.248
26.167
N3
3n
f1 (ppm)

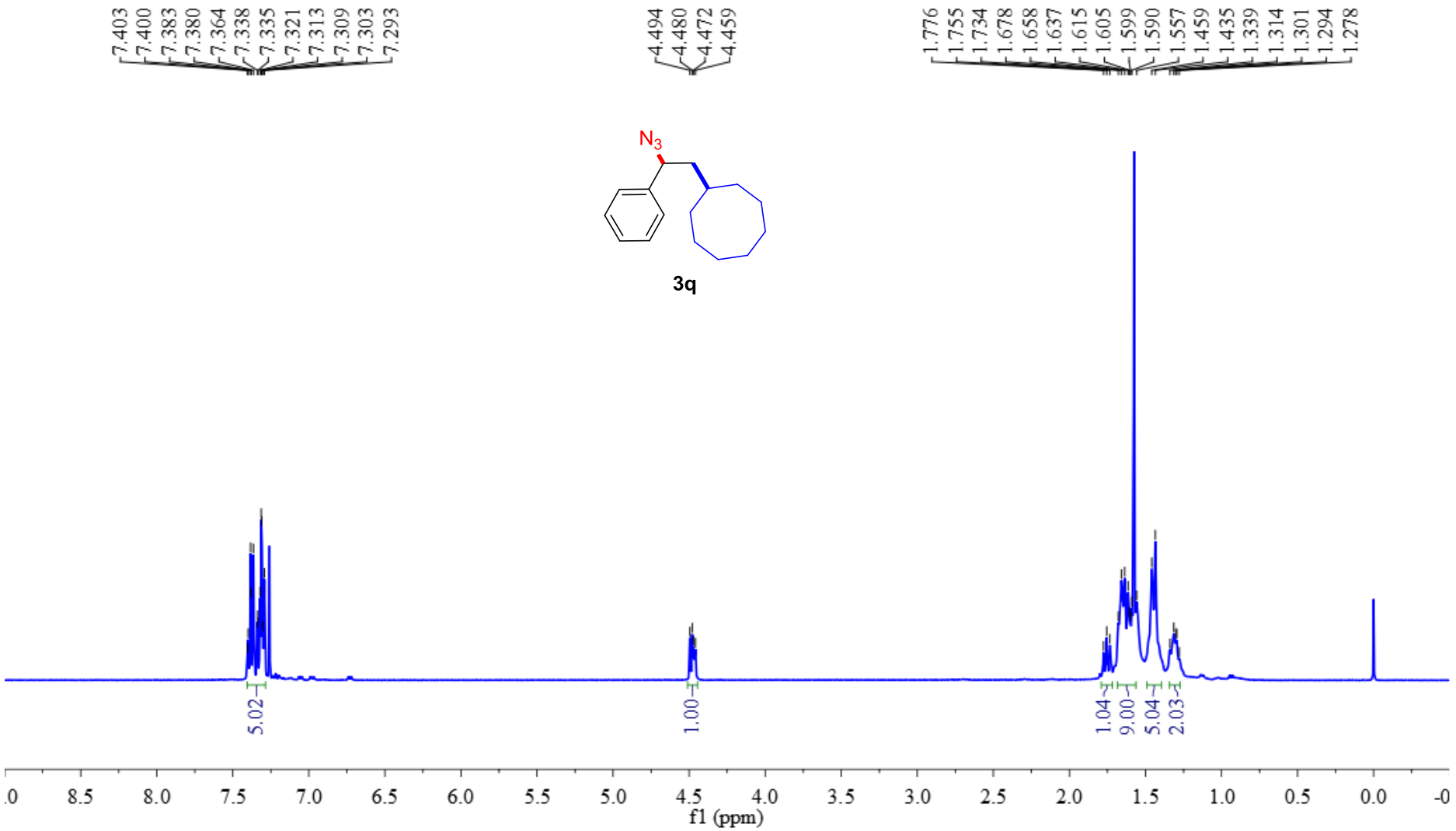

7.414
7.410
7.393
7.375
7.351
7.347
7.333
7.326
7.322
7.306
4.446
4.429
4.411
1.893
1.874
1.830
1.814
1.800
1.788
1.780
1.763
1.751
1.734
1.640
1.631
1.622
1.616
1.602
1.582
1.537
1.527
1.517
1.508
1.498
1.489
1.156
1.148
1.139
1.129
1.119
N3
3o
5.00
1.00
1.00
4.04
2.00
2.01
2.01
f1 (ppm)

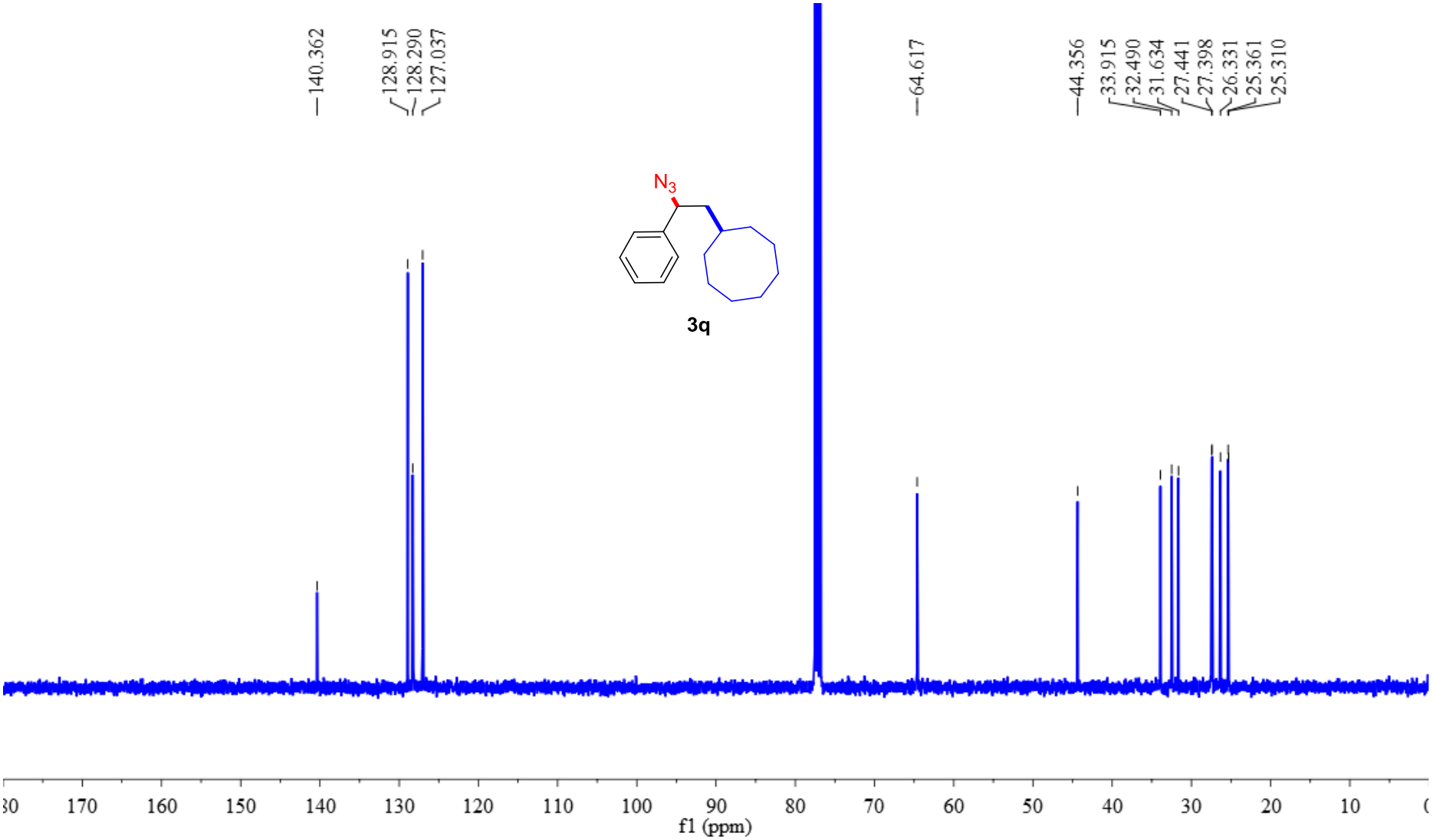

140.130
128.884
128.310
127.061
65.894
42.549
36.991
32.664
25.203
25.108
N3
3o
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


7.398
7.386
7.382
7.378
7.366
7.363
7.337
7.334
7.320
7.310
7.305
7.301
7.289
4.485
4.471
4.464
4.449
1.790
1.770
1.768
1.757
1.749
1.742
1.735
1.724
1.715
1.638
1.623
1.619
1.604
1.593
1.586
1.574
1.544
1.541
1.498
1.487
1.483
1.478
1.475
1.470
1.458
1.452
1.449
1.445
1.434
1.430
1.426
1.423
1.402
1.395
1.246
1.240
1.229
1.223
1.218
1.212
1.207
N3
5.01
1.00
3.01
6.02
4.02
2.00
f1 (ppm)

3p


144.061
128.498
127.219
125.640
67.115
49.330
34.758
34.473
34.042
26.849
26.390
26.299
N3
3p
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


7.403
7.400
7.383
7.380
7.364
7.338
7.335
7.321
7.313
7.309
7.303
7.293
4.494
4.480
4.472
4.459
1.776
1.755
1.734
1.678
1.658
1.637
1.615
1.605
1.599
1.590
1.557
1.459
1.435
1.339
1.314
1.301
1.294
1.278
N3
3q
5.02
1.00
1.04
9.00
5.04
2.03
f1 (ppm)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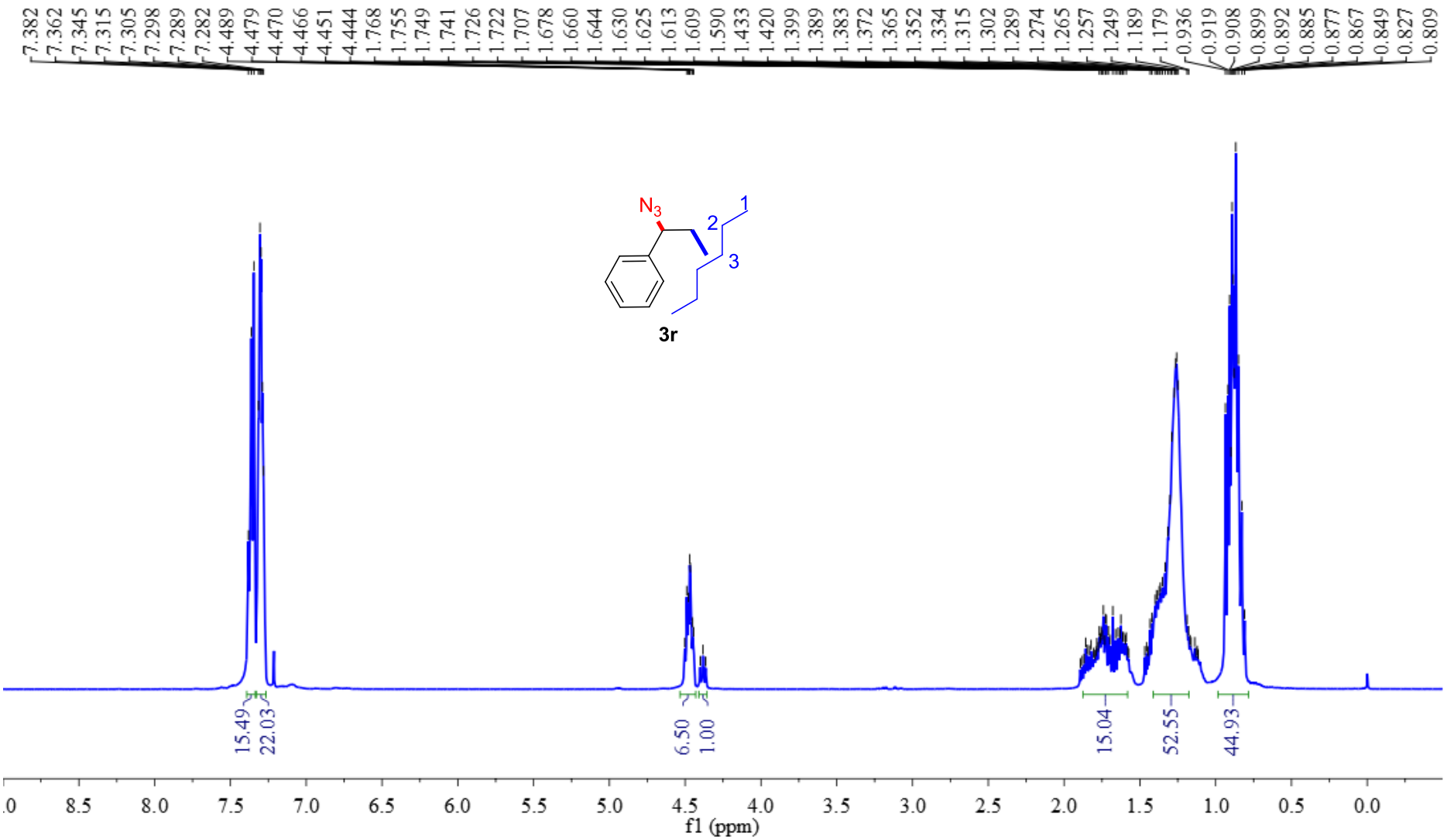
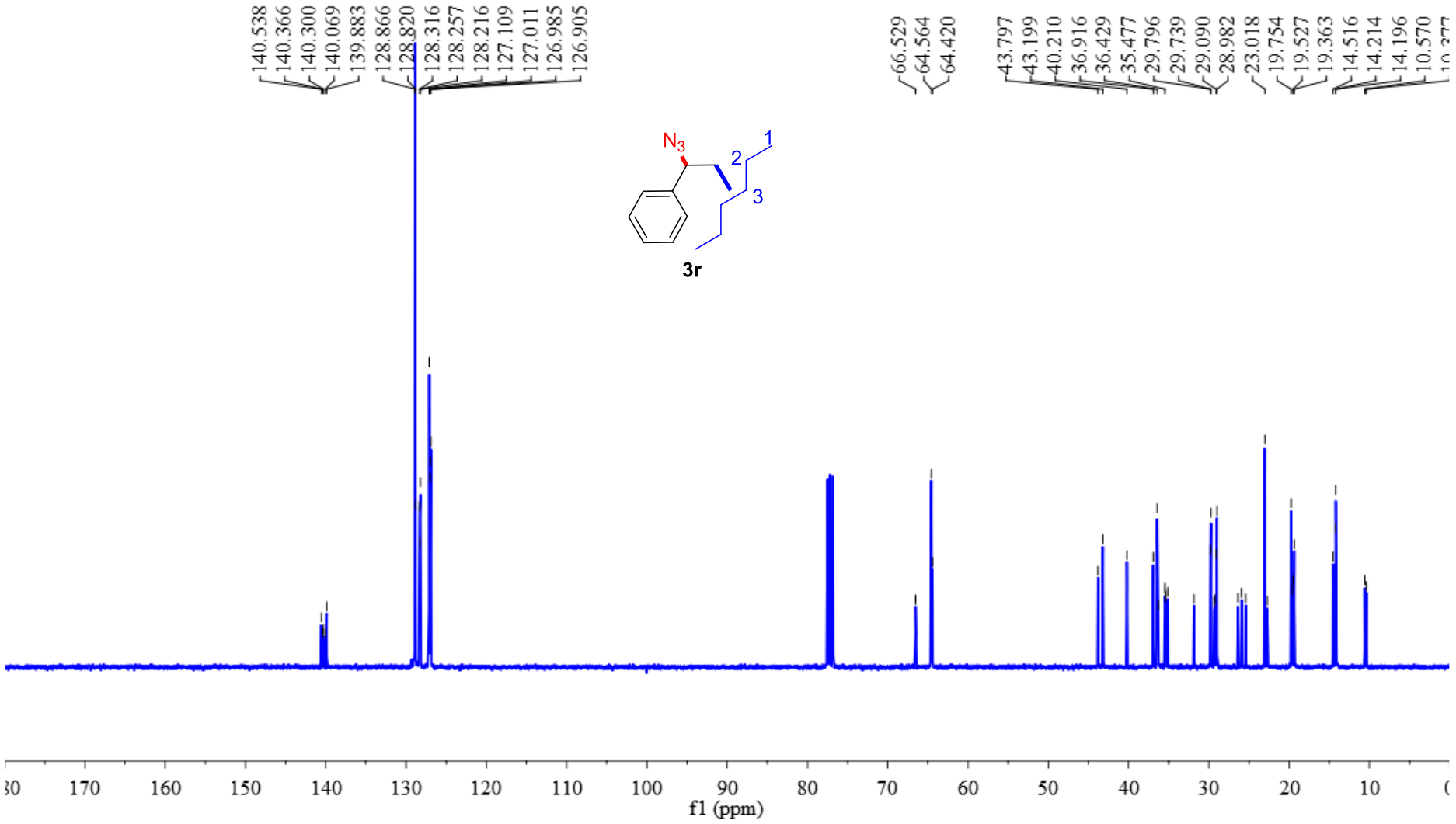
128.915
128.290
127.037
64.617
44.356
33.915
32.490
31.634
27.441
27.398
26.331
25.361
25.310
N3
3q
f1 (ppm)


7.382
7.362
7.345
7.315
7.305
7.298
7.289
7.282
4.489
4.479
4.470
4.466
4.451
4.444
1.768
1.755
1.749
1.741
1.726
1.722
1.707
1.678
1.660
1.644
1.630
1.625
1.613
1.609
1.590
1.433
1.420
1.399
1.389
1.383
1.372
1.365
1.352
1.334
1.315
1.302
1.289
1.274
1.265
1.257
1.249
1.189
1.179
0.936
0.919
0.908
0.899
0.892
0.885
0.877
0.867
0.849
0.827
0.809
N3
1
2
3
3r
15.49
22.03
6.50
1.00
15.04
52.55
44.93
f1 (ppm)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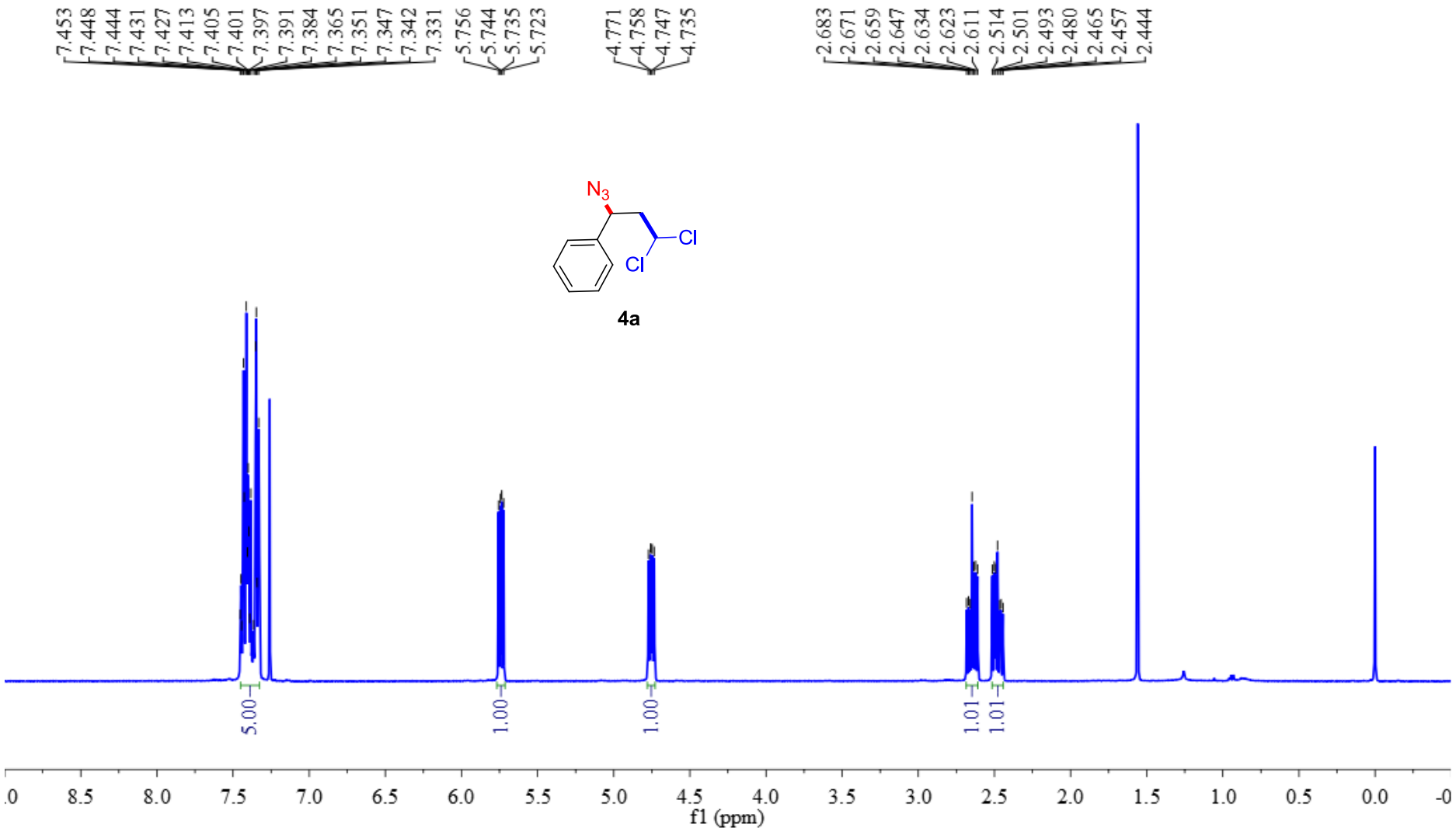
140.366
140.300
140.069
139.883
128.866
128.820
128.316
128.257
128.216
127.109
127.011
126.985
126.905
66.529
64.564
64.420
43.797
43.199
40.210
36.916
36.429
35.477
29.796
29.739
29.090
28.982
23.018
19.754
19.527
19.363
14.516
14.214
14.196
10.570
N3
1
2
3
3r
f1 (ppm)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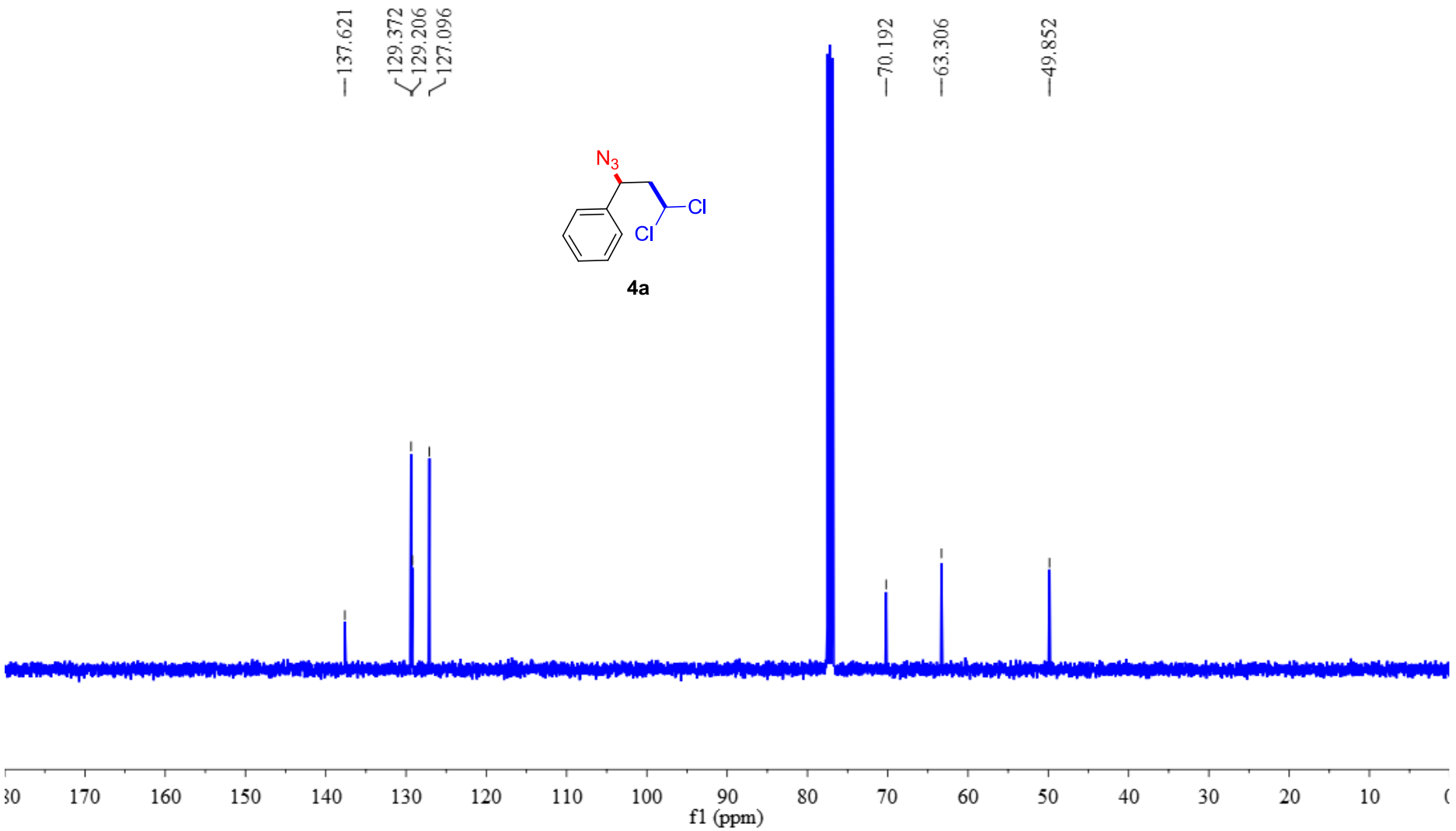
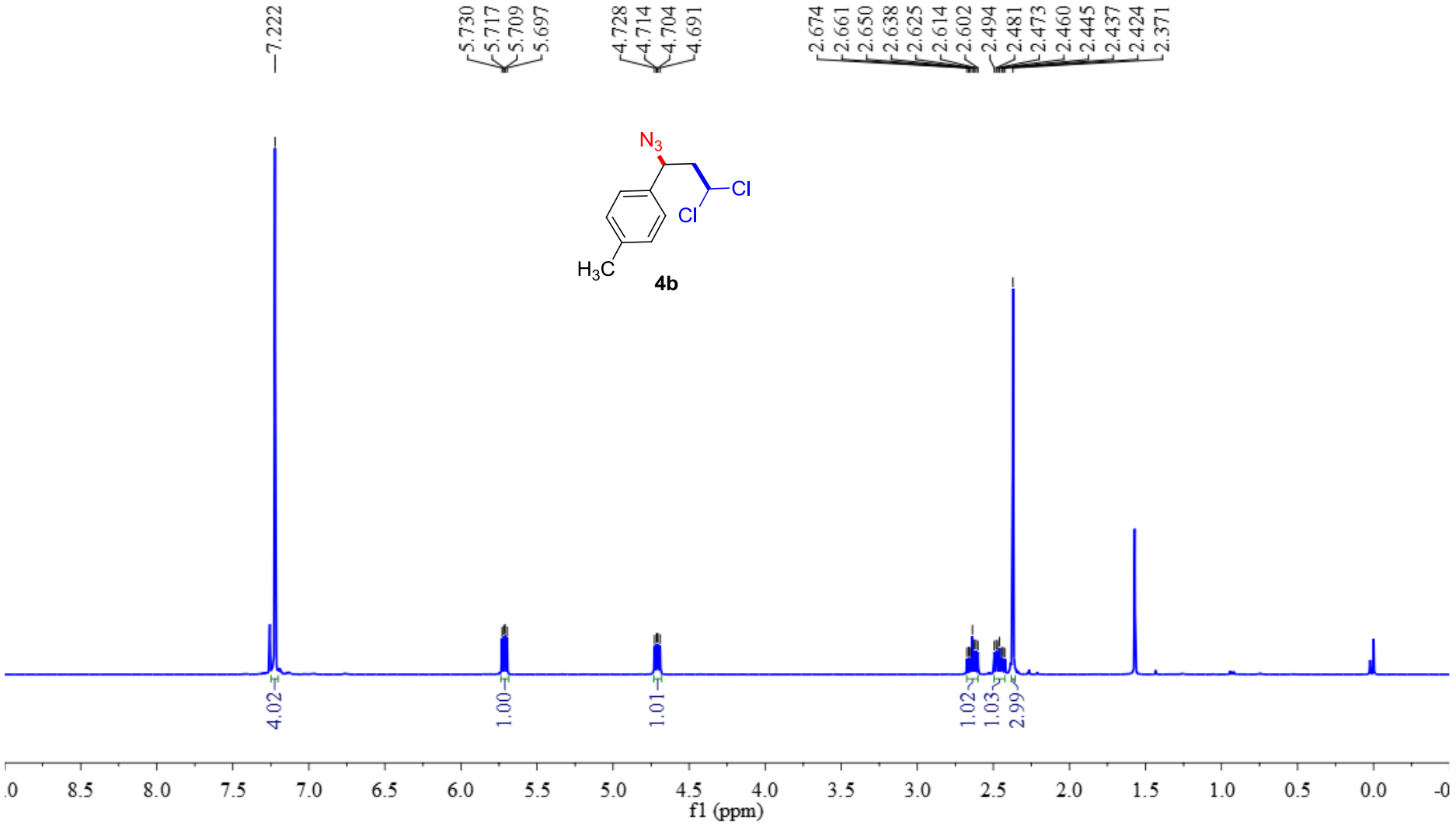
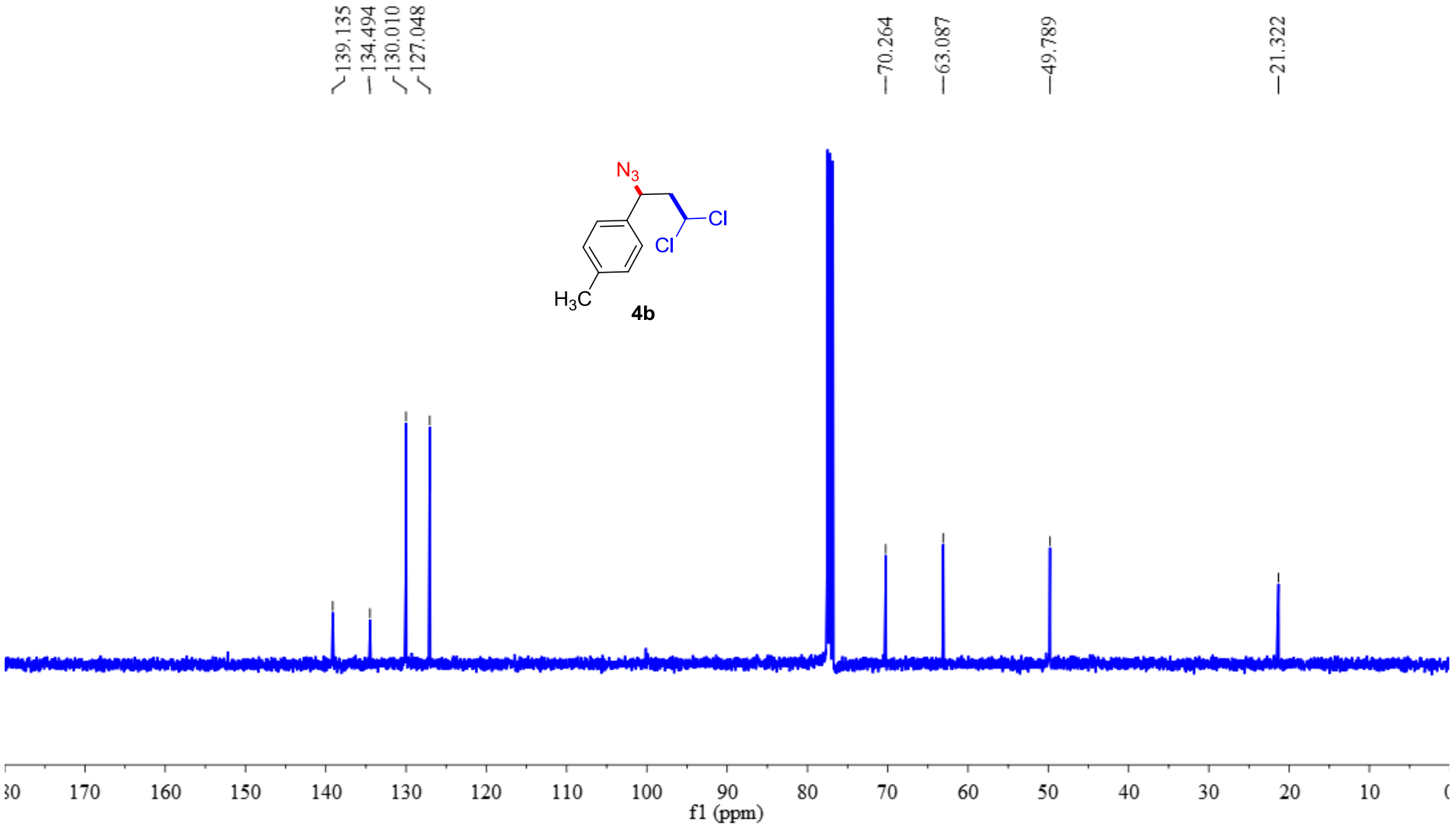
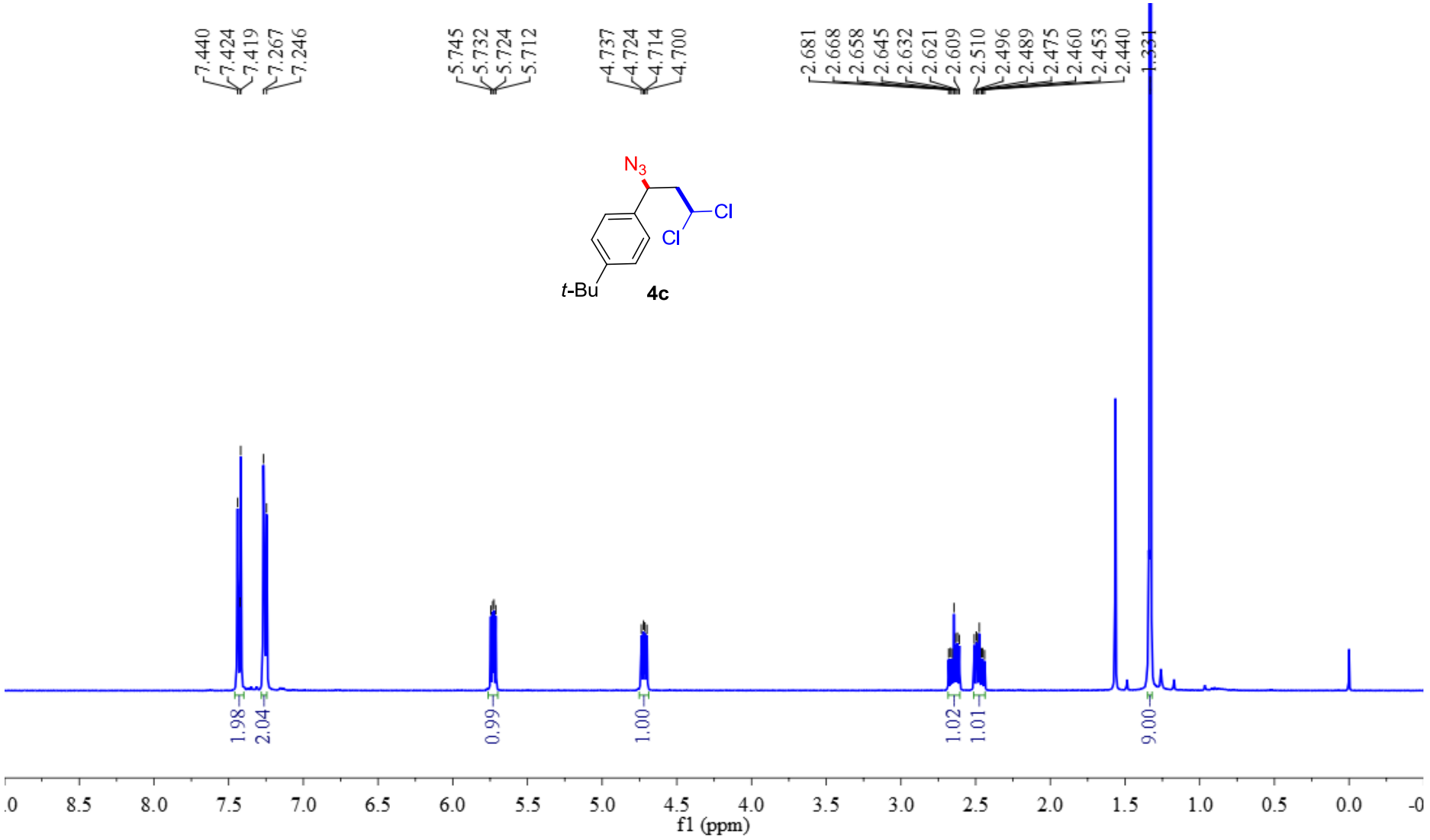
7.448
7.444
7.431
7.427
7.413
7.405
7.401
7.397
7.391
7.384
7.365
7.351
7.347
7.342
7.331
5.756
5.744
5.735
5.723
4.771
4.758
4.747
4.735
2.683
2.671
2.659
2.647
2.634
2.623
2.611
2.514
2.501
2.493
2.480
2.465
2.457
2.444
N3
Cl
Cl
4a
5.00
1.00
1.00
1.01
1.01
f1 (ppm)


137.621
129.372
129.206
127.096
70.192
63.306
49.852
N3
Cl
Cl
4a
f1 (ppm)


7.222
5.730
5.717
5.709
5.697
4.728
4.714
4.704
4.691
2.674
2.661
2.650
2.638
2.625
2.614
2.602
2.494
2.481
2.473
2.460
2.445
2.437
2.424
2.371
N3
Cl
Cl
H3C
4b
4.02
1.00
1.01
1.02
1.03
2.99
f1 (ppm)


139.135
134.494
130.010
127.048
70.264
63.087
49.789
21.322
N3
Cl
Cl
H3C
4b
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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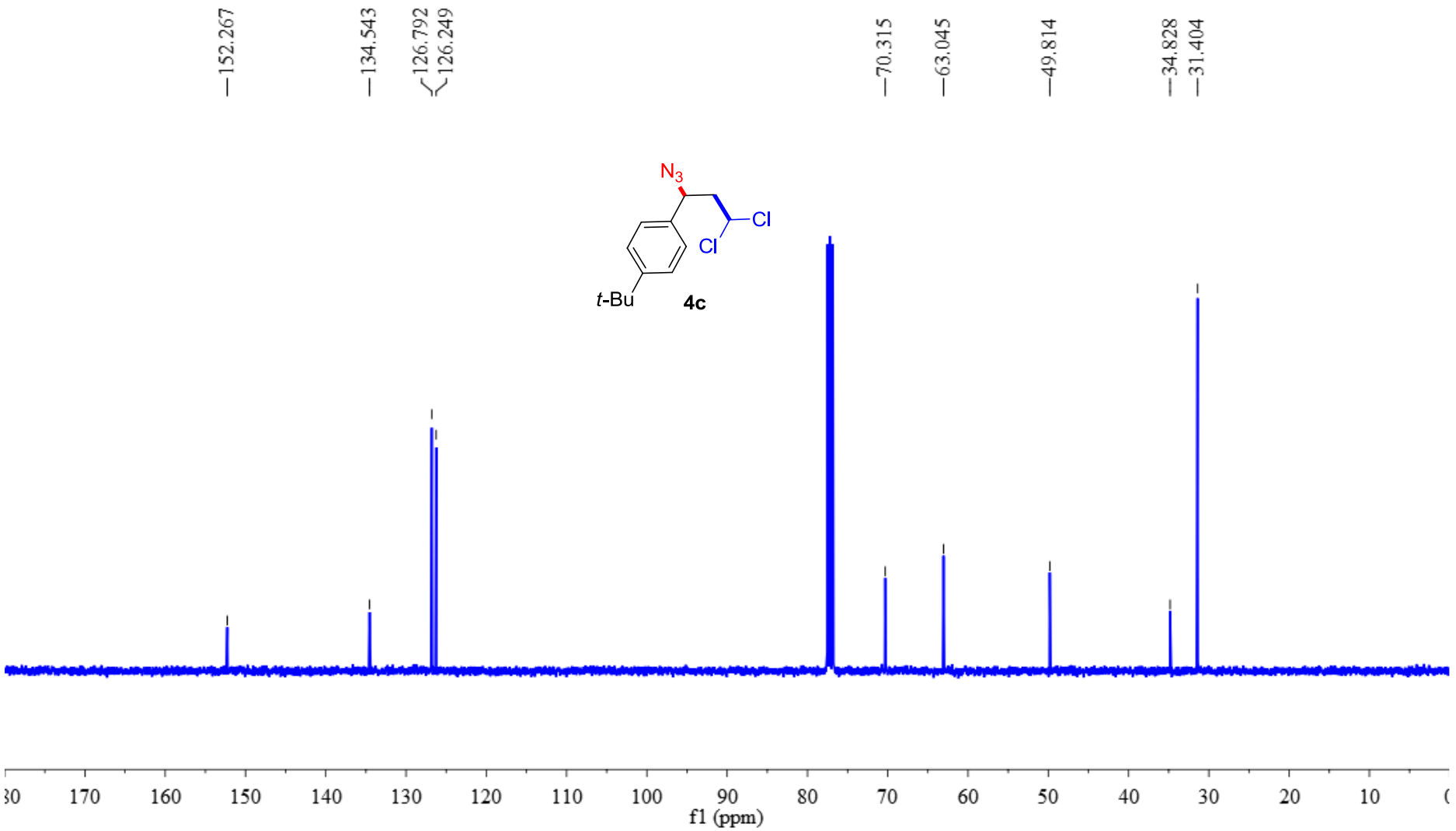
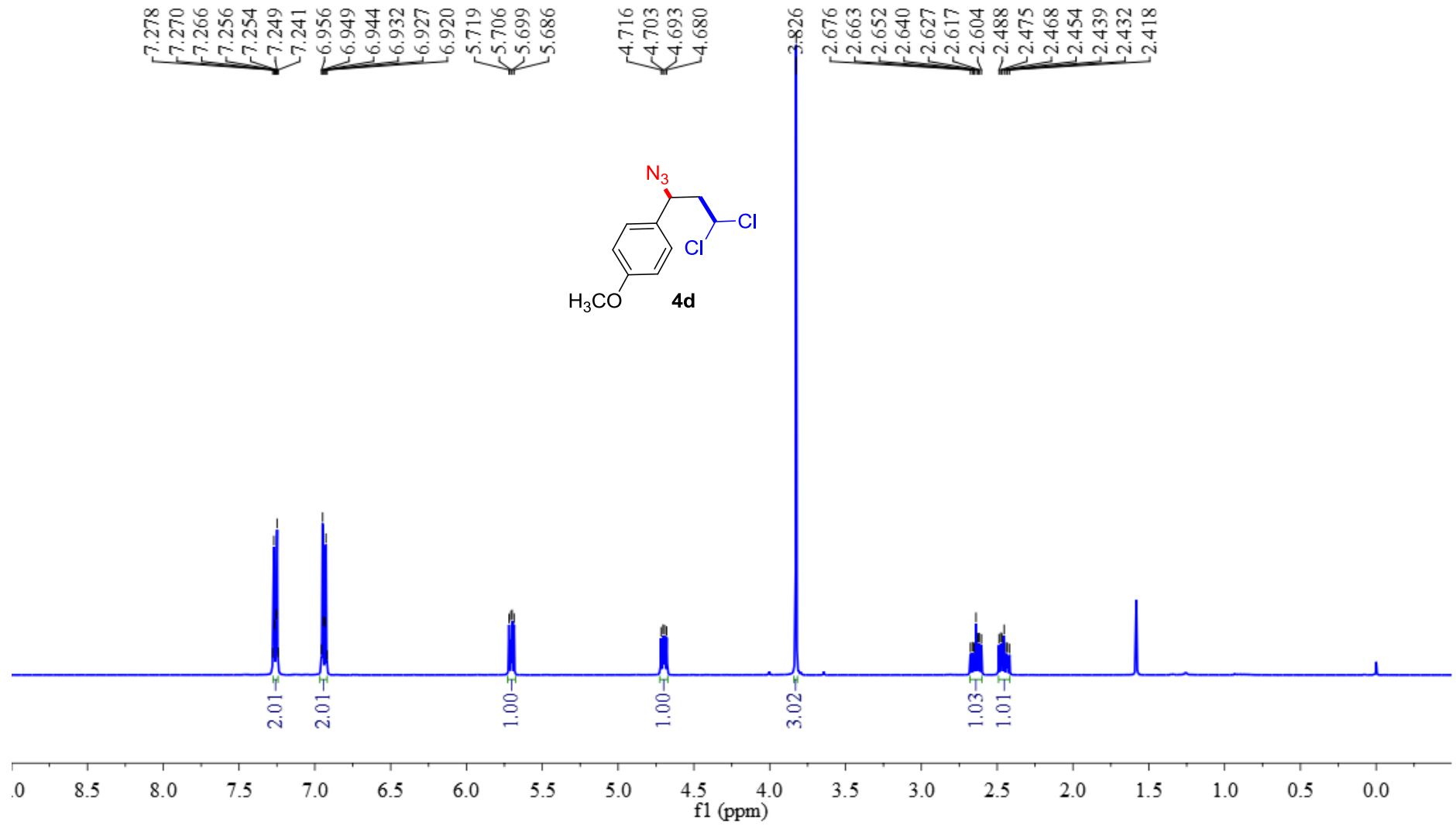
7.424
7.419
7.267
7.246
5.745
5.732
5.724
5.712
4.737
4.724
4.714
4.700
2.681
2.668
2.658
2.645
2.632
2.621
2.609
2.510
2.496
2.489
2.475
2.460
2.453
2.440
1.331
N3
Cl
Cl
t-Bu
4c
1.98
2.04
0.99
1.00
1.02
1.01
9.00
f1 (ppm)


152.267
134.543
126.792
126.249
70.315
63.045
49.814
34.828
31.404
N3
Cl
Cl
t-Bu
4c
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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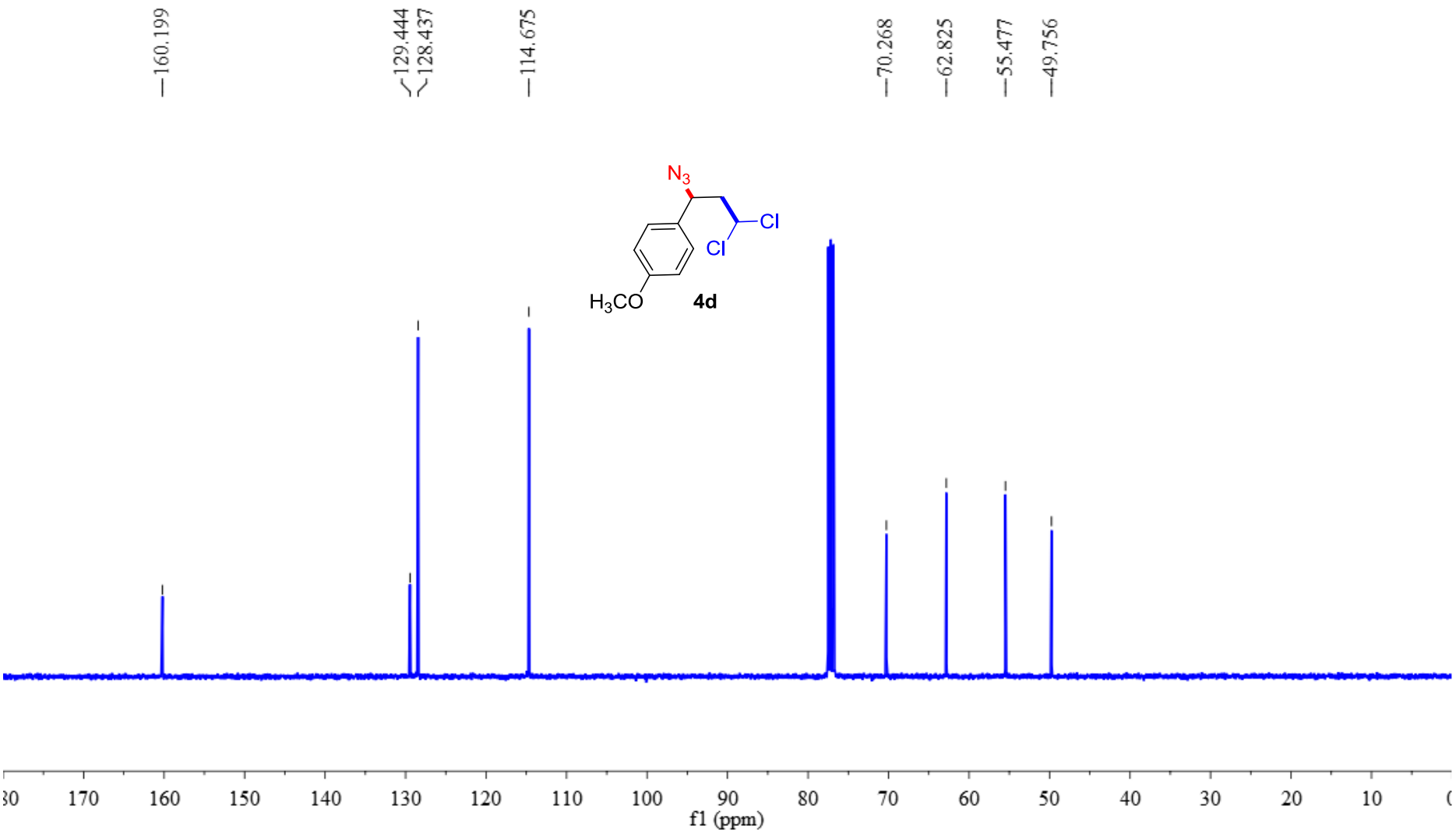
7.270
7.266
7.256
7.254
7.249
7.241
6.956
6.949
6.944
6.932
6.927
6.920
5.719
5.706
5.699
5.686
4.716
4.703
4.693
4.680
3.826
2.676
2.663
2.652
2.640
2.627
2.617
2.604
2.488
2.475
2.468
2.454
2.439
2.432
2.418
N3
Cl
Cl
H3CO
4d
2.01
2.01
1.00
1.00
3.02
1.03
1.01
f1 (ppm)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129.444
128.437
114.675
70.268
62.825
55.477
49.756
N3
Cl
Cl
H3CO
4d
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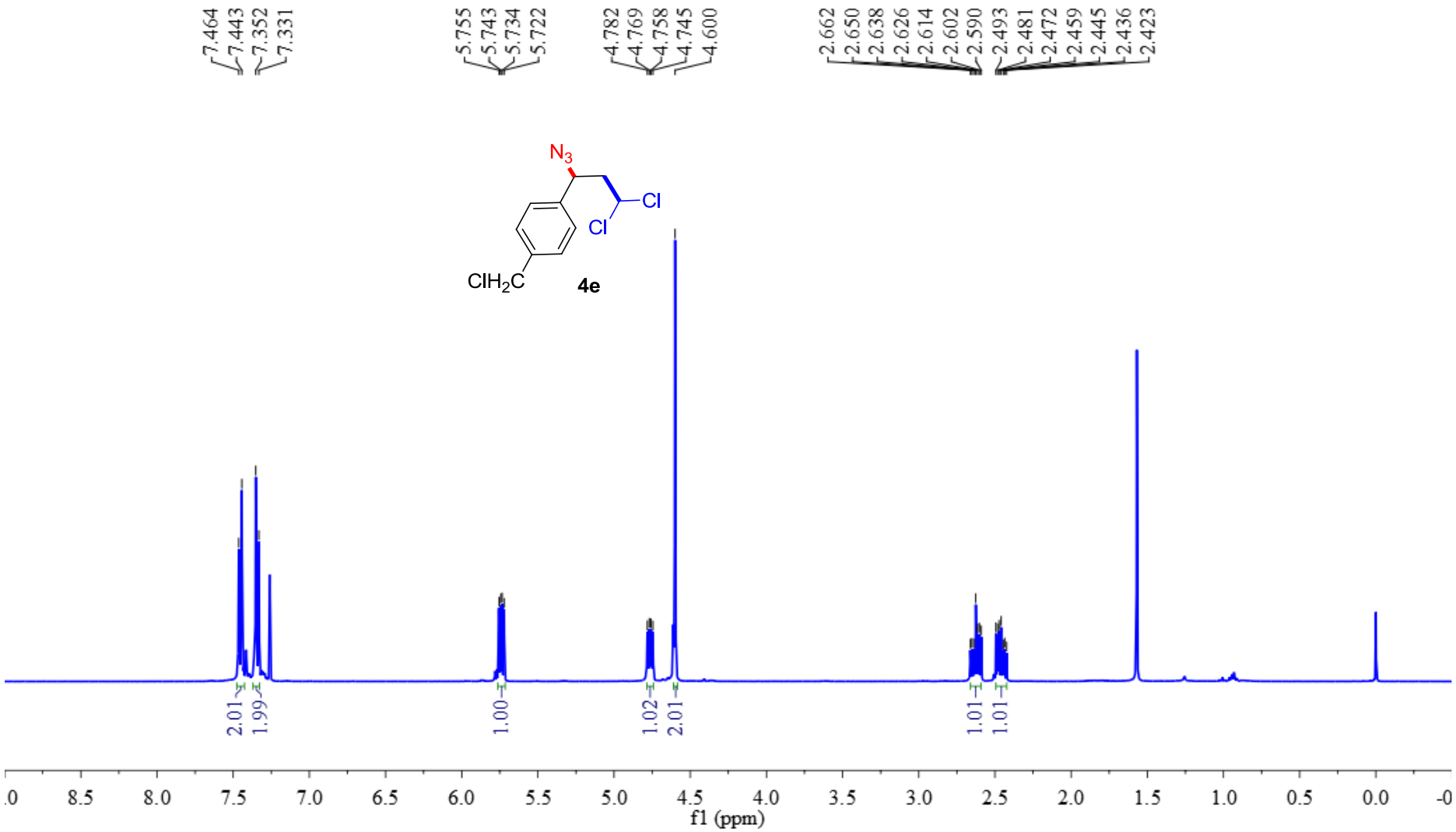
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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7.443
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7.331
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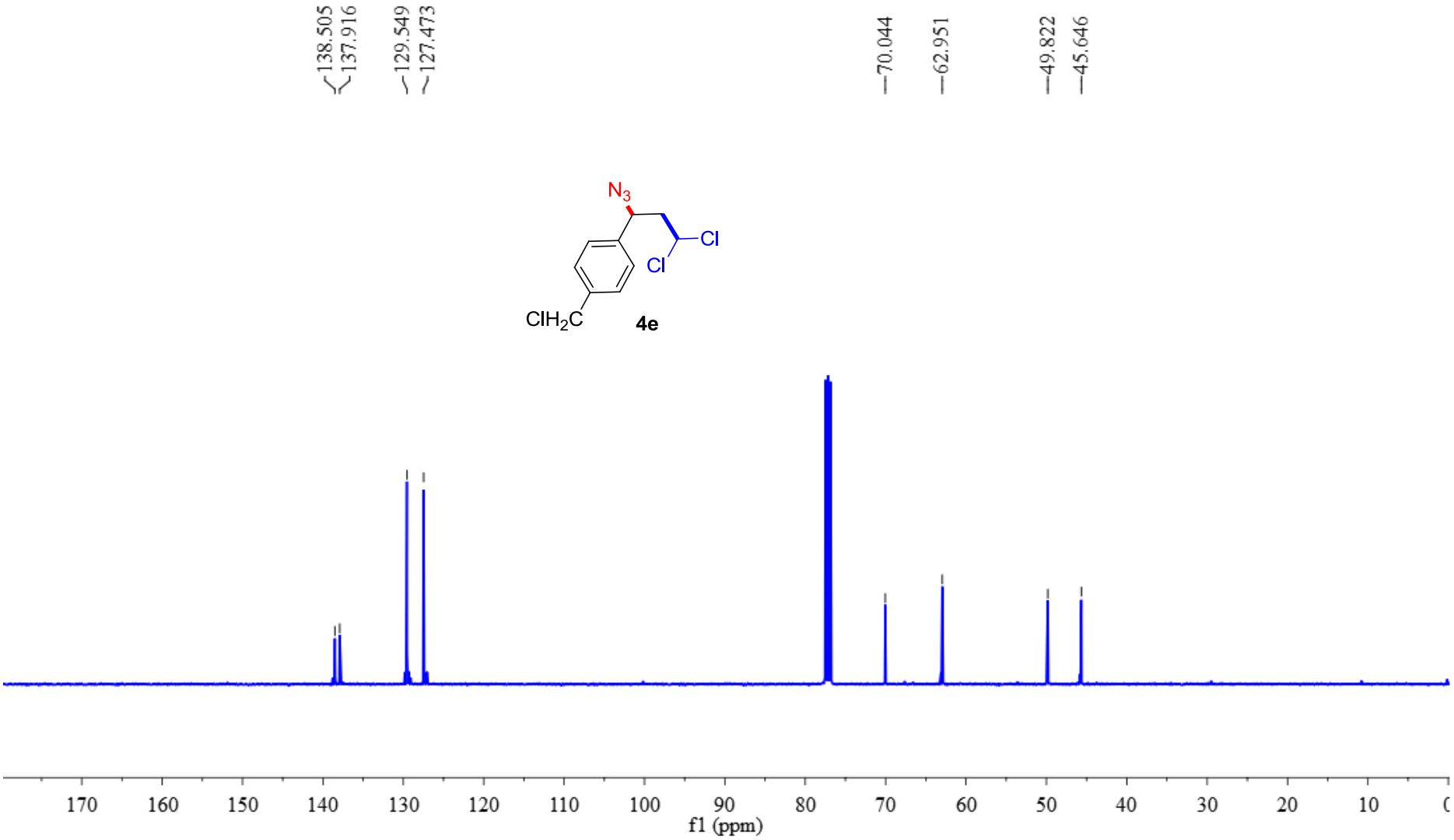
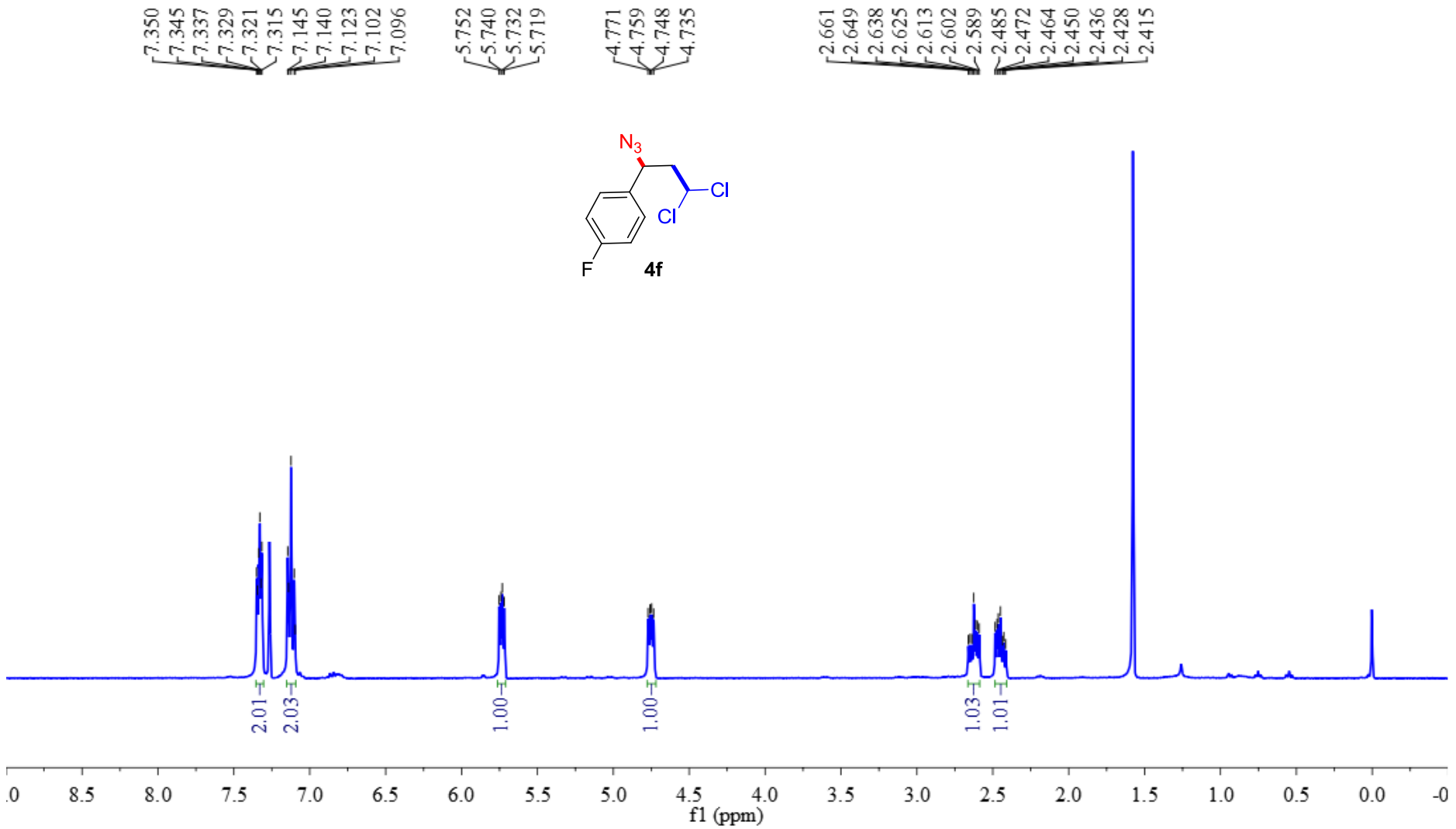
5.743
5.734
5.722
4.782
4.769
4.758
4.745
4.600
2.662
2.650
2.638
2.626
2.614
2.602
2.590
2.493
2.481
2.472
2.459
2.445
2.436
2.423
N3
Cl
Cl
ClH2C
4e
2.01
1.99
1.00
1.02
2.01
1.01
1.01
f1 (ppm)


138.505
137.916
129.549
127.473
70.044
62.951
49.822
45.646
N3
Cl
Cl
ClH2C
4e
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


7.350
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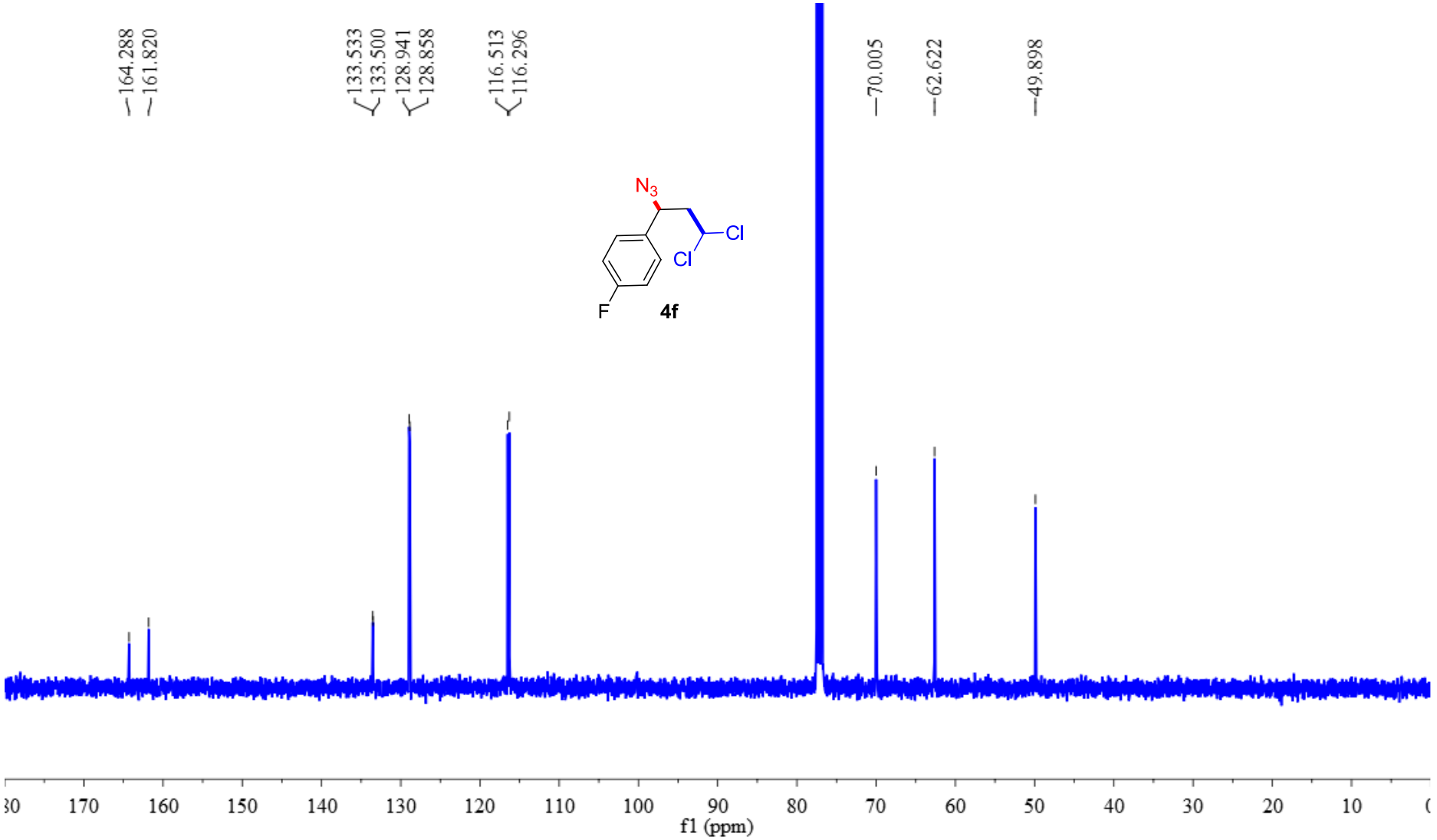
7.337
7.329
7.321
7.315
7.145
7.140
7.123
7.102
7.096
5.752
5.740
5.732
5.719
4.771
4.759
4.748
4.735
2.661
2.649
2.638
2.625
2.613
2.602
2.589
2.485
2.472
2.464
2.450
2.436
2.428
2.415
N3
Cl
Cl
F
4f
2.01
2.03
1.00
1.00
1.03
1.01
f1 (ppm)


164.288
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133.500
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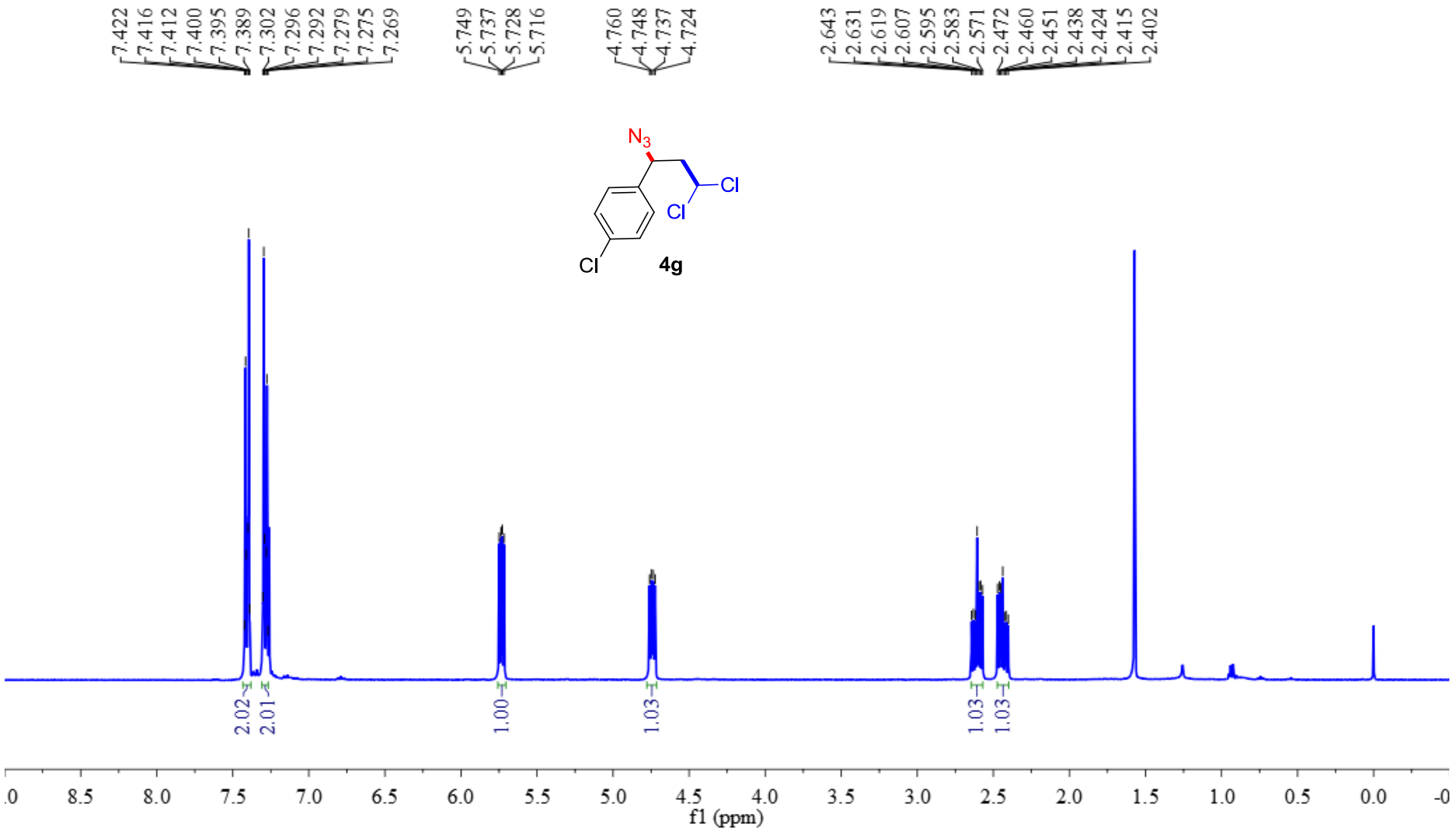
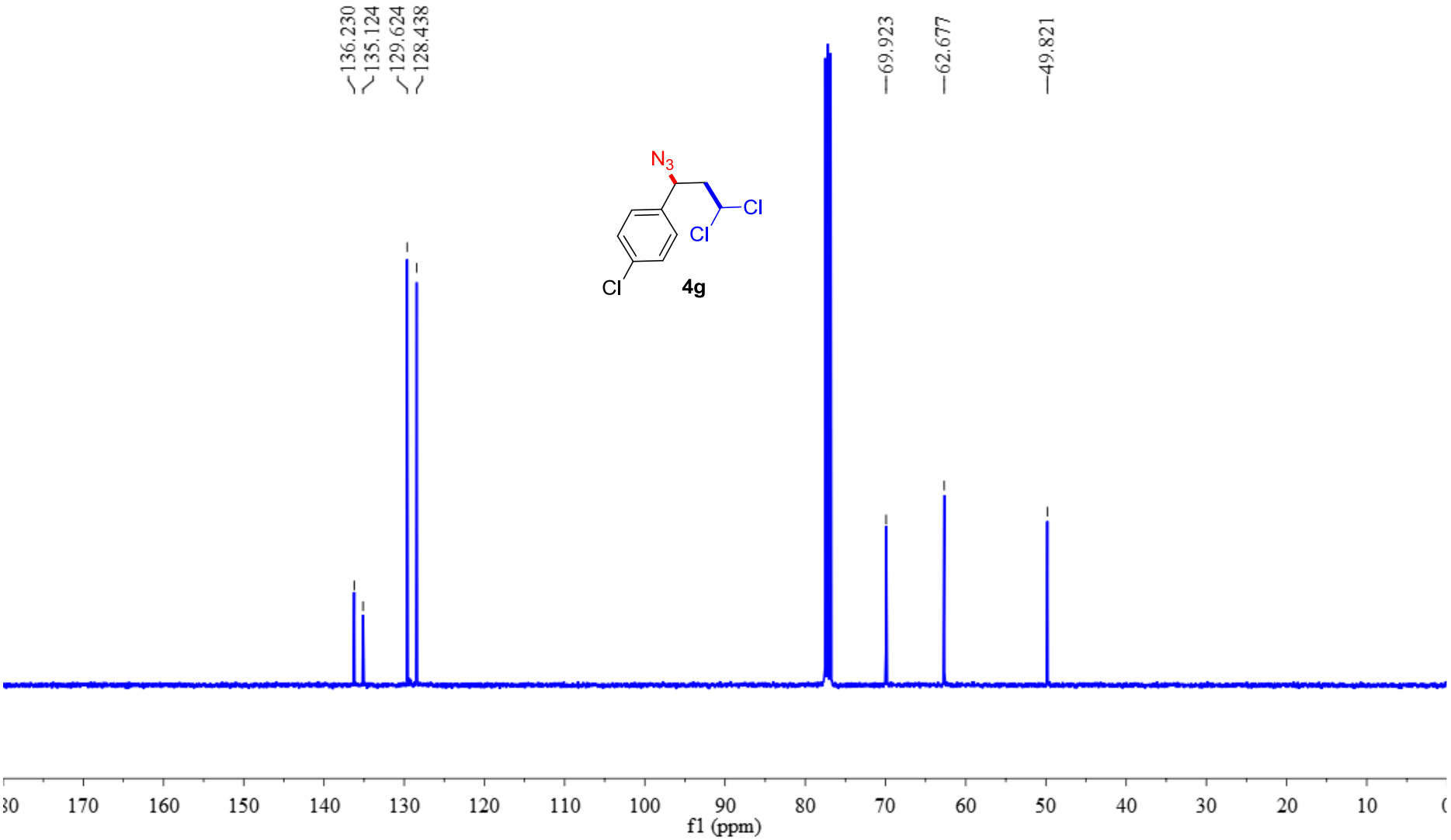
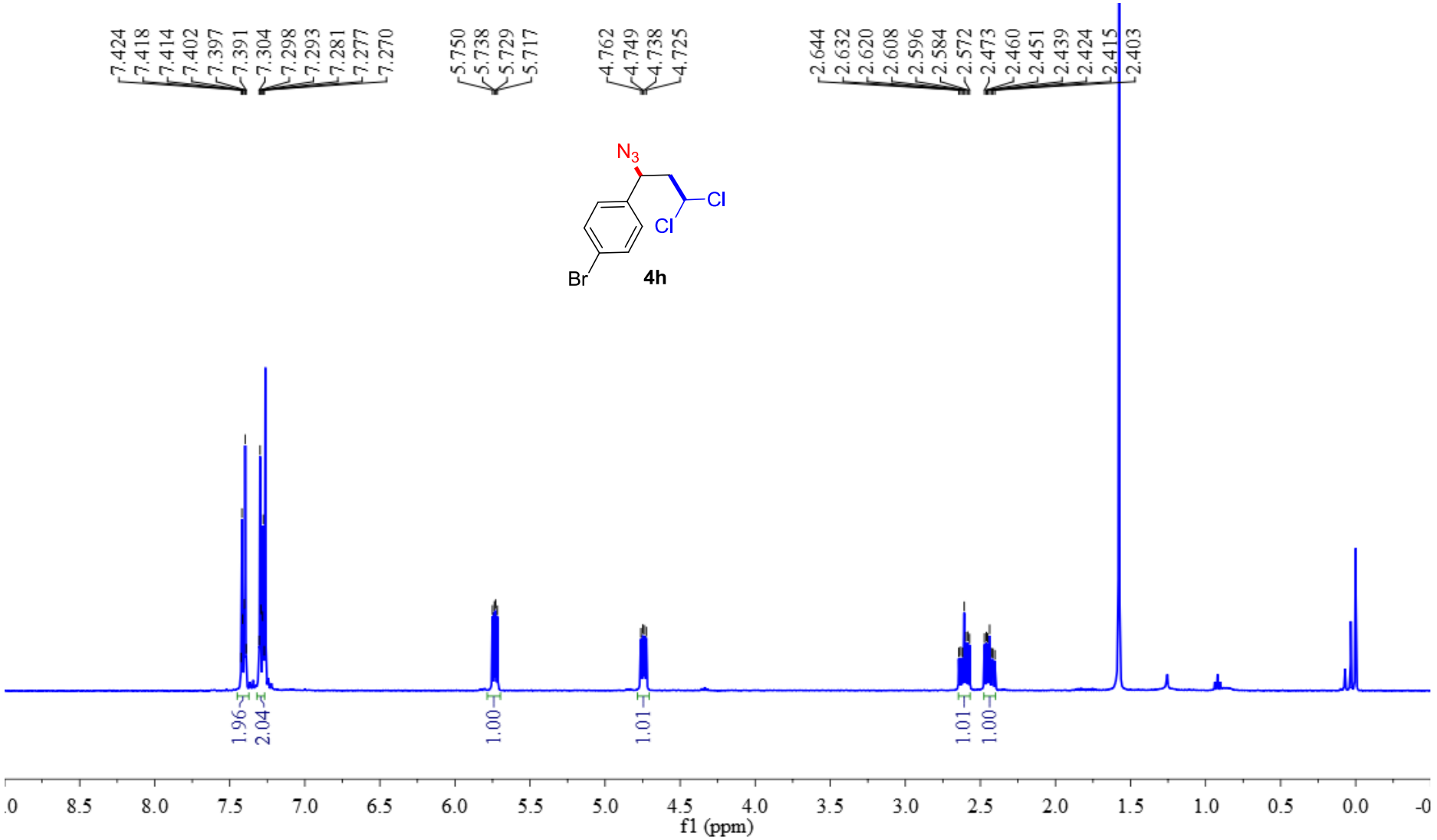
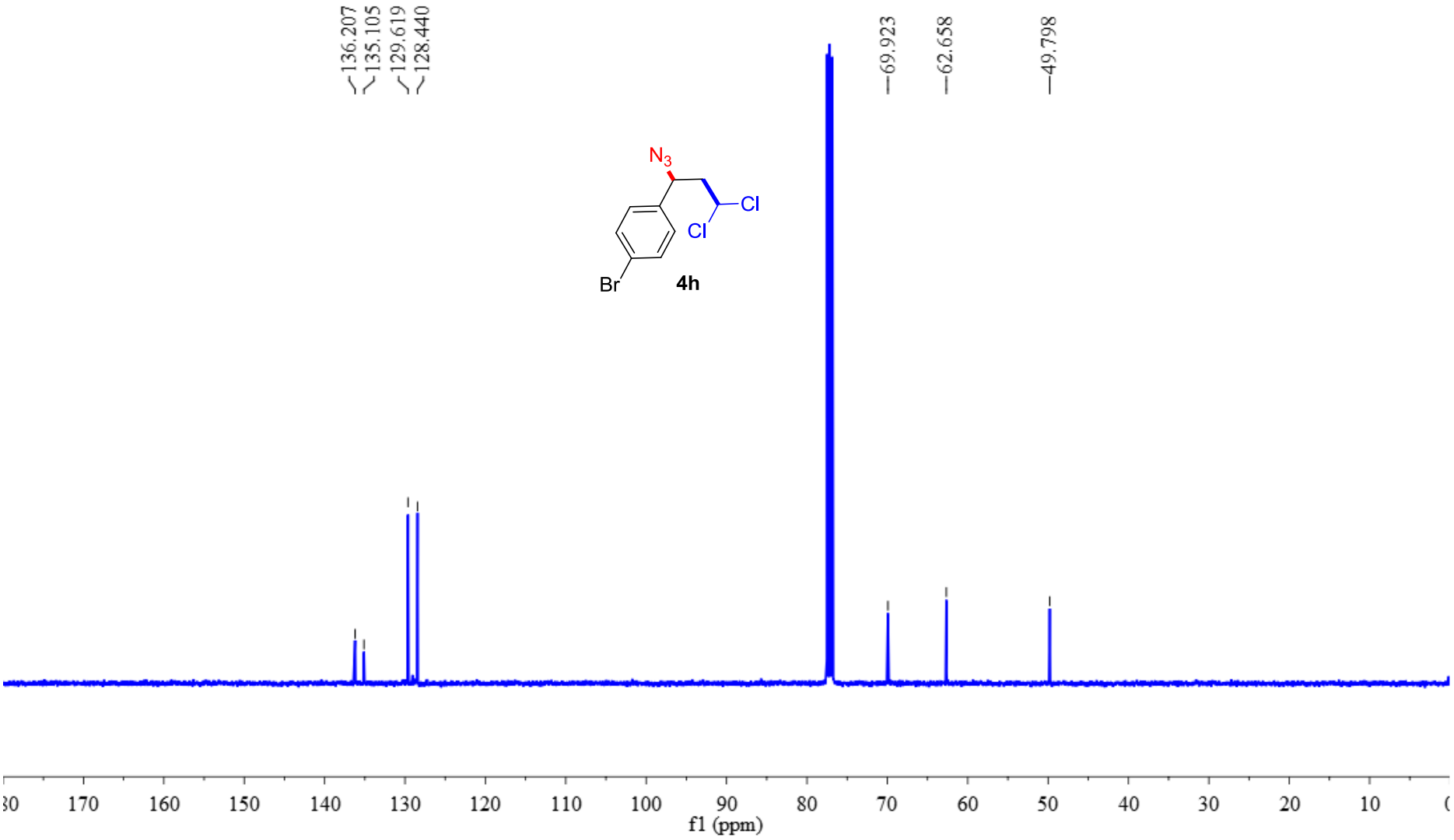
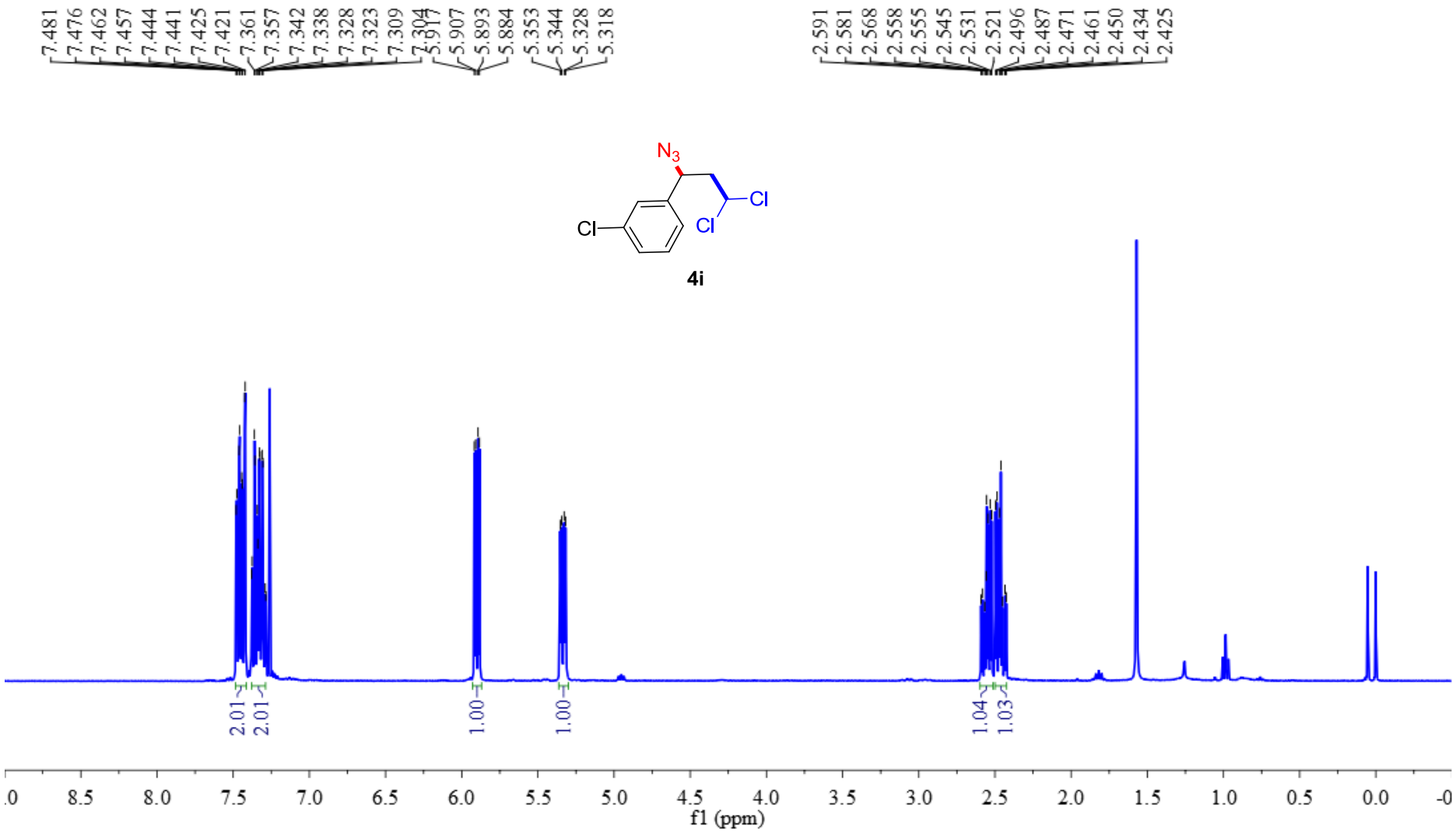
116.513
116.296
70.005
62.622
49.898
N3
Cl
Cl
F
4f
f1 (ppm)

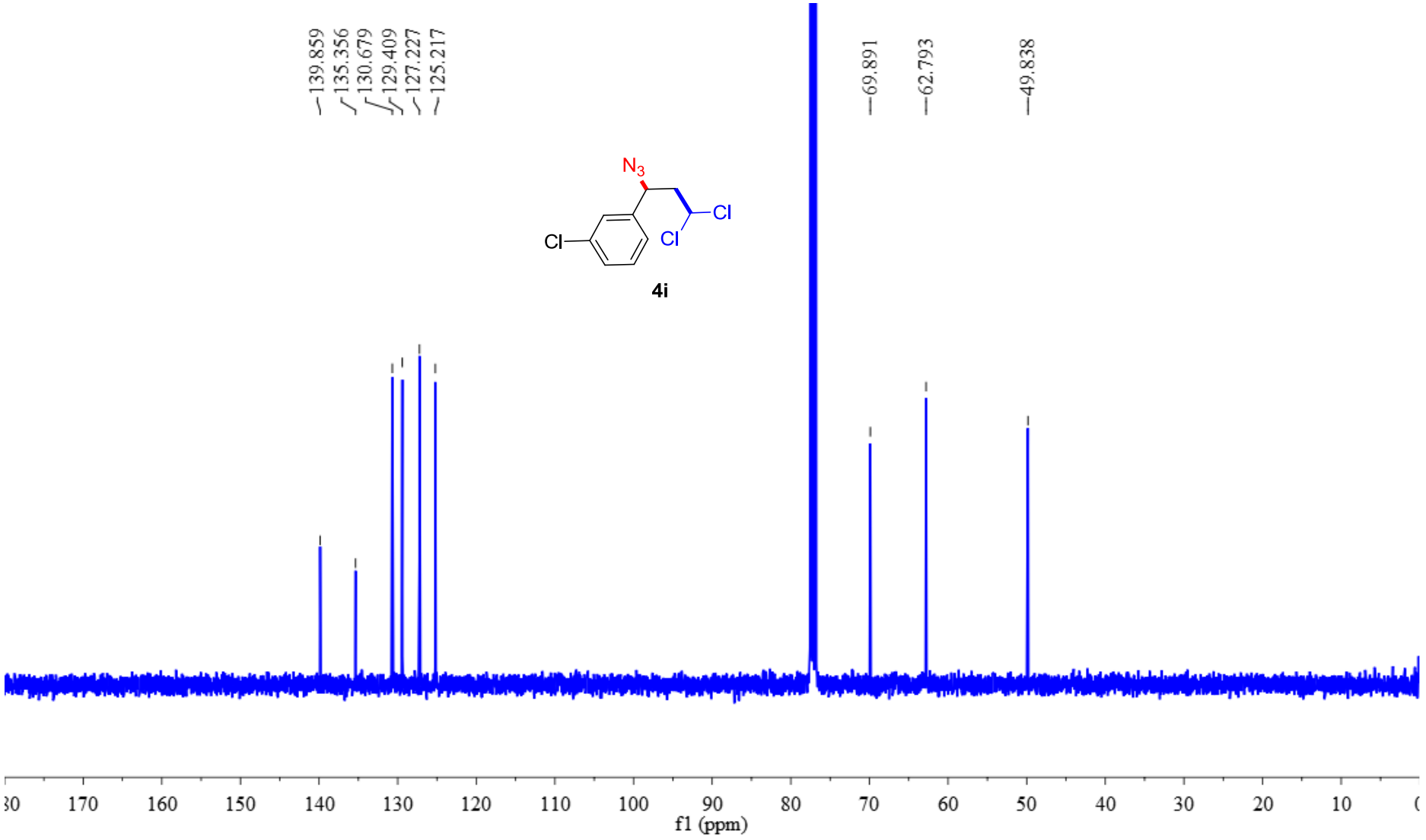

7.422
7.416
7.412
7.400
7.395
7.389
7.302
7.296
7.292
7.279
7.275
7.269
5.749
5.737
5.728
5.716
4.760
4.748
4.737
4.724
2.643
2.631
2.619
2.607
2.595
2.583
2.571
2.472
2.460
2.451
2.438
2.424
2.415
2.402
N3
Cl
Cl
Cl
4g
2.02
2.01
1.00
1.03
1.03
1.03
f1 (ppm)

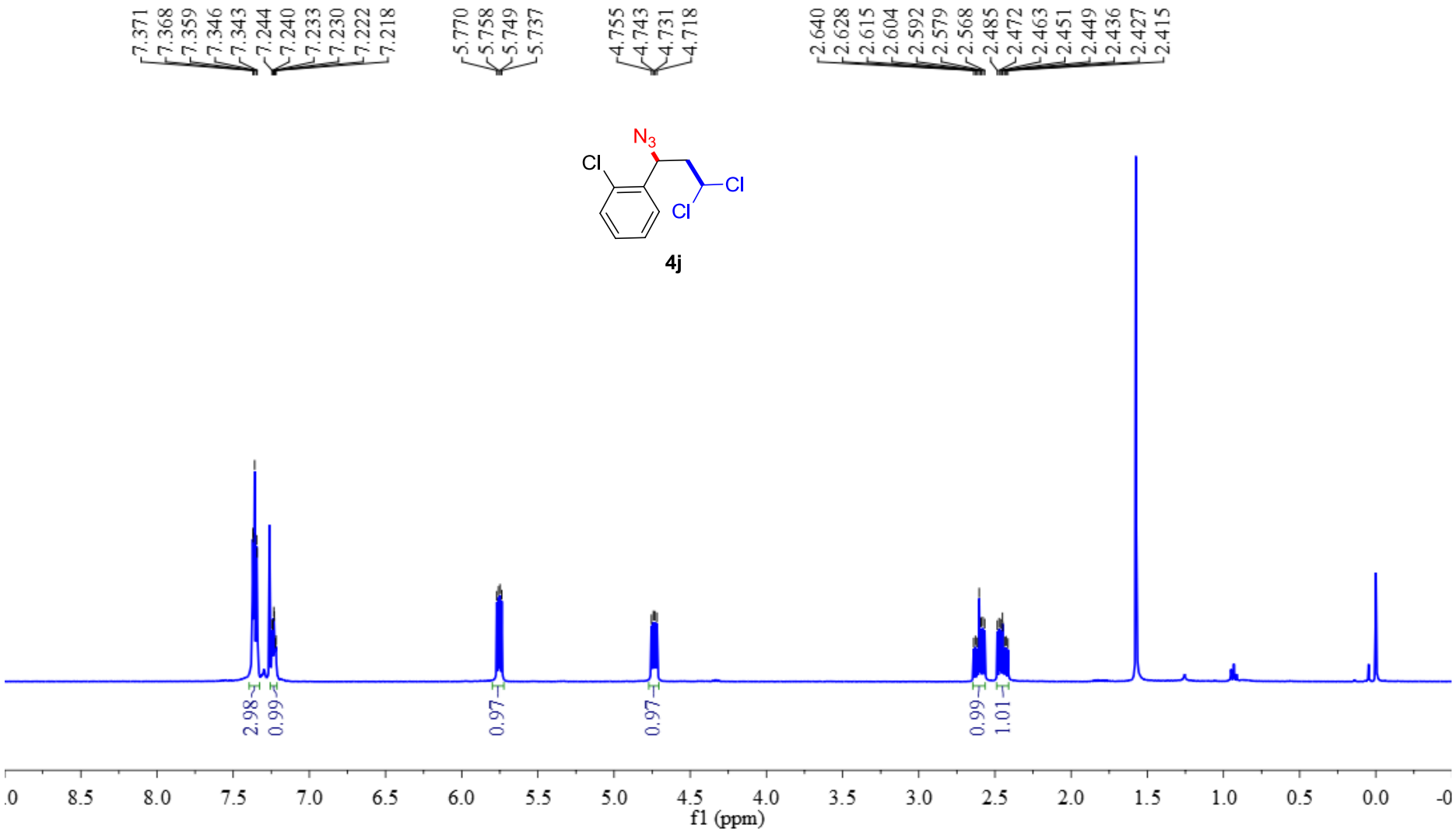

136.230
135.124
129.624
128.438
69.923
62.677
49.821
N3
Cl
Cl
Cl
4g
f1 (ppm)


7.424
7.418
7.414
7.402
7.397
7.391
7.304
7.298
7.293
7.281
7.277
7.270
5.750
5.738
5.729
5.717
4.762
4.749
4.738
4.725
2.644
2.632
2.620
2.608
2.596
2.584
2.572
2.473
2.460
2.451
2.439
2.424
2.415
2.403
N3
Cl
Cl
Br
4h
1.96
2.04
1.00
1.01
1.01
1.00
f1 (ppm)


136.207
135.105
129.619
128.440
69.923
62.658
49.798
N3
Cl
Cl
Br
4h
f1 (ppm)


7.481
7.476
7.462
7.457
7.444
7.441
7.425
7.421
7.361
7.357
7.342
7.338
7.328
7.323
7.309
7.304
5.917
5.907
5.893
5.884
5.353
5.344
5.328
5.318
2.591
2.581
2.568
2.558
2.555
2.545
2.531
2.521
2.496
2.487
2.471
2.461
2.450
2.434
2.425
N3
Cl
Cl
Cl
4i
2.01
2.01
1.00
1.00
1.04
1.03
f1 (ppm)


139.859
135.356
130.679
129.409
127.227
125.217
69.891
62.793
49.838
N3
Cl
Cl
Cl
4i
f1 (ppm)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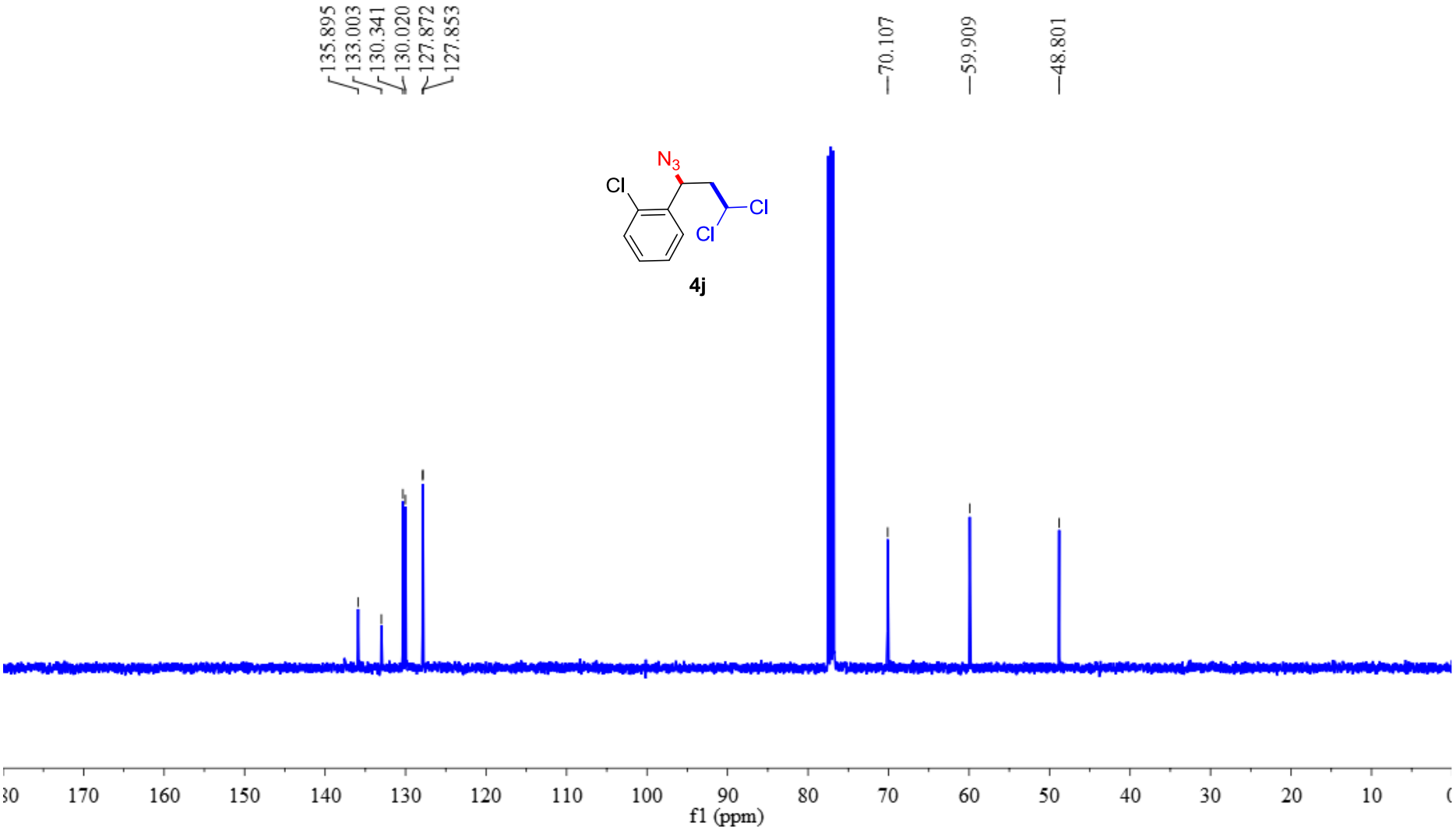
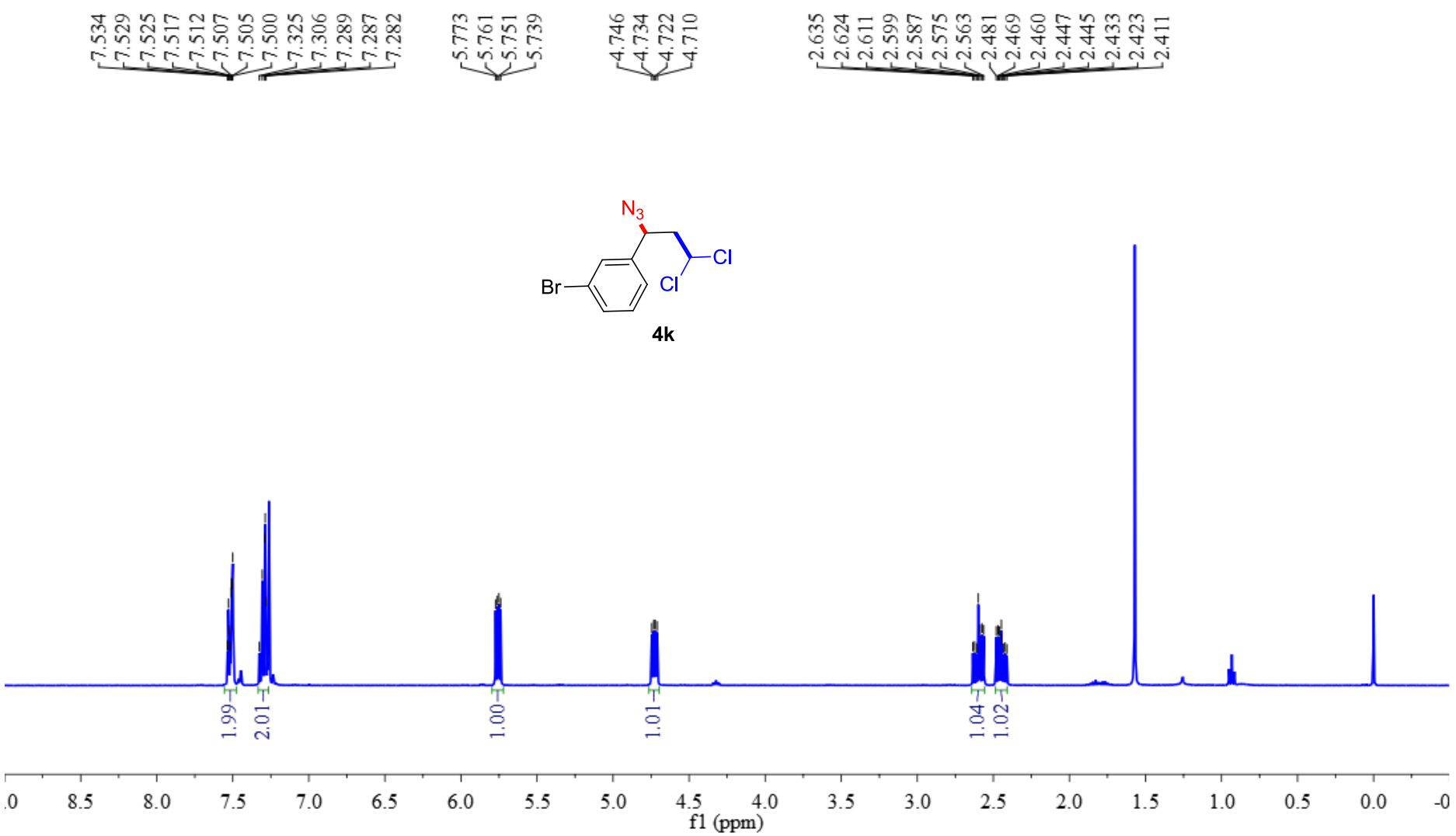
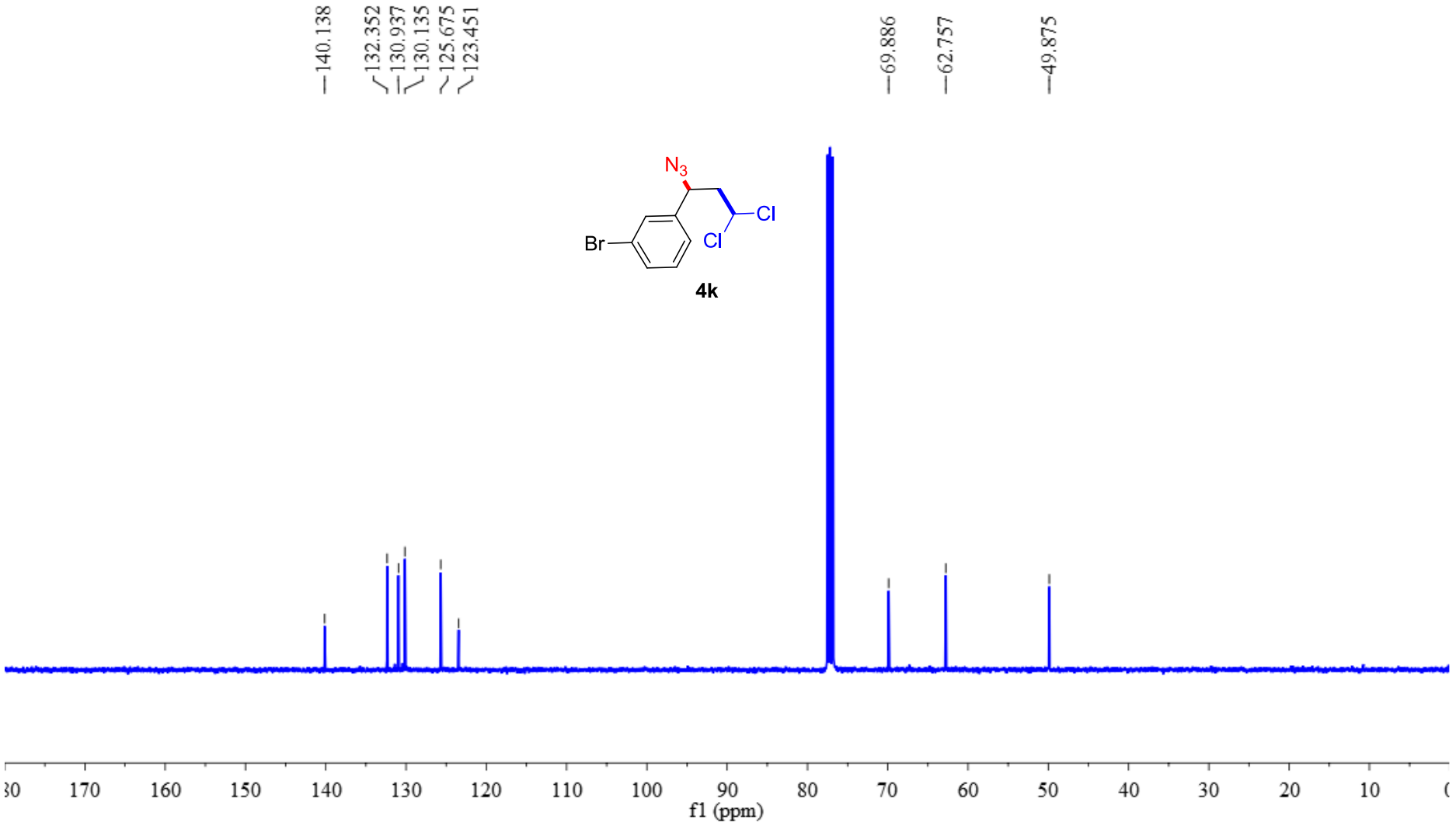
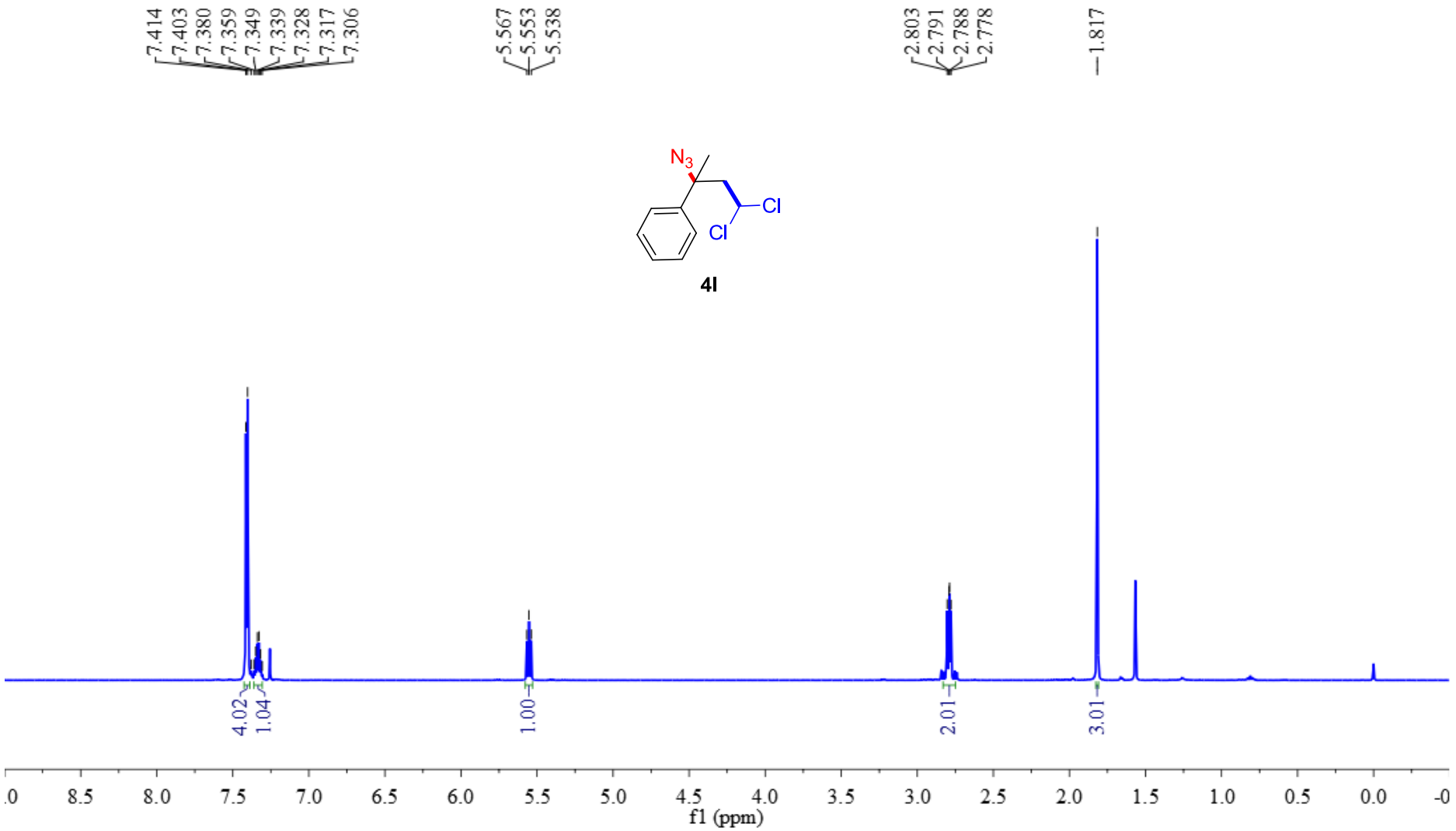
7.368
7.359
7.346
7.343
7.244
7.240
7.233
7.230
7.222
7.218
5.770
5.758
5.749
5.737
4.755
4.743
4.731
4.718
2.640
2.628
2.615
2.604
2.592
2.579
2.568
2.485
2.472
2.463
2.451
2.449
2.436
2.427
2.415
N3
Cl
Cl
Cl
4j
2.98
0.99
0.97
0.97
0.99
1.01
f1 (ppm)

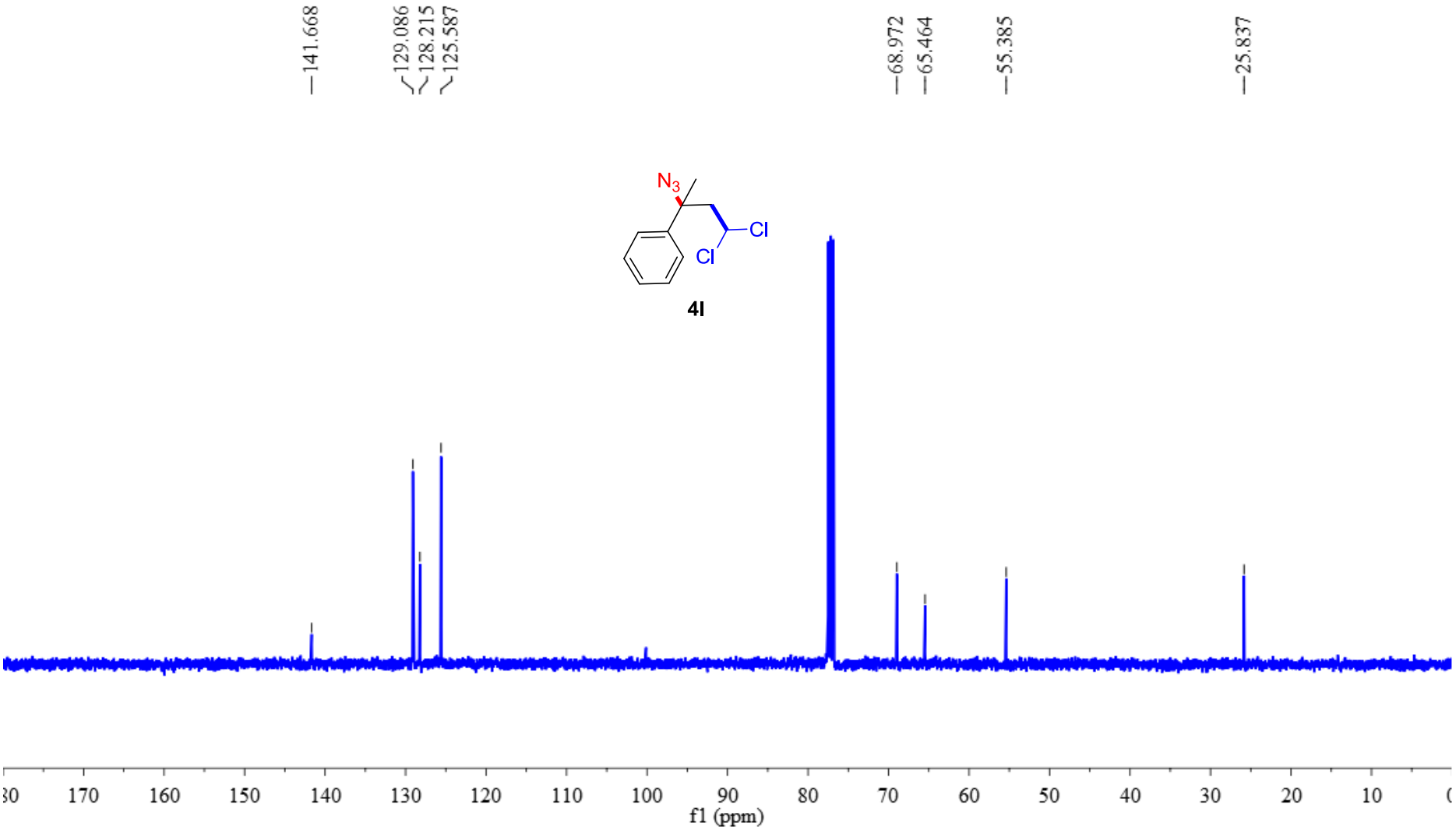

135.895
133.003
130.341
130.020
127.872
127.853
70.107
59.909
48.801
N3
Cl
Cl
Cl
4j
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

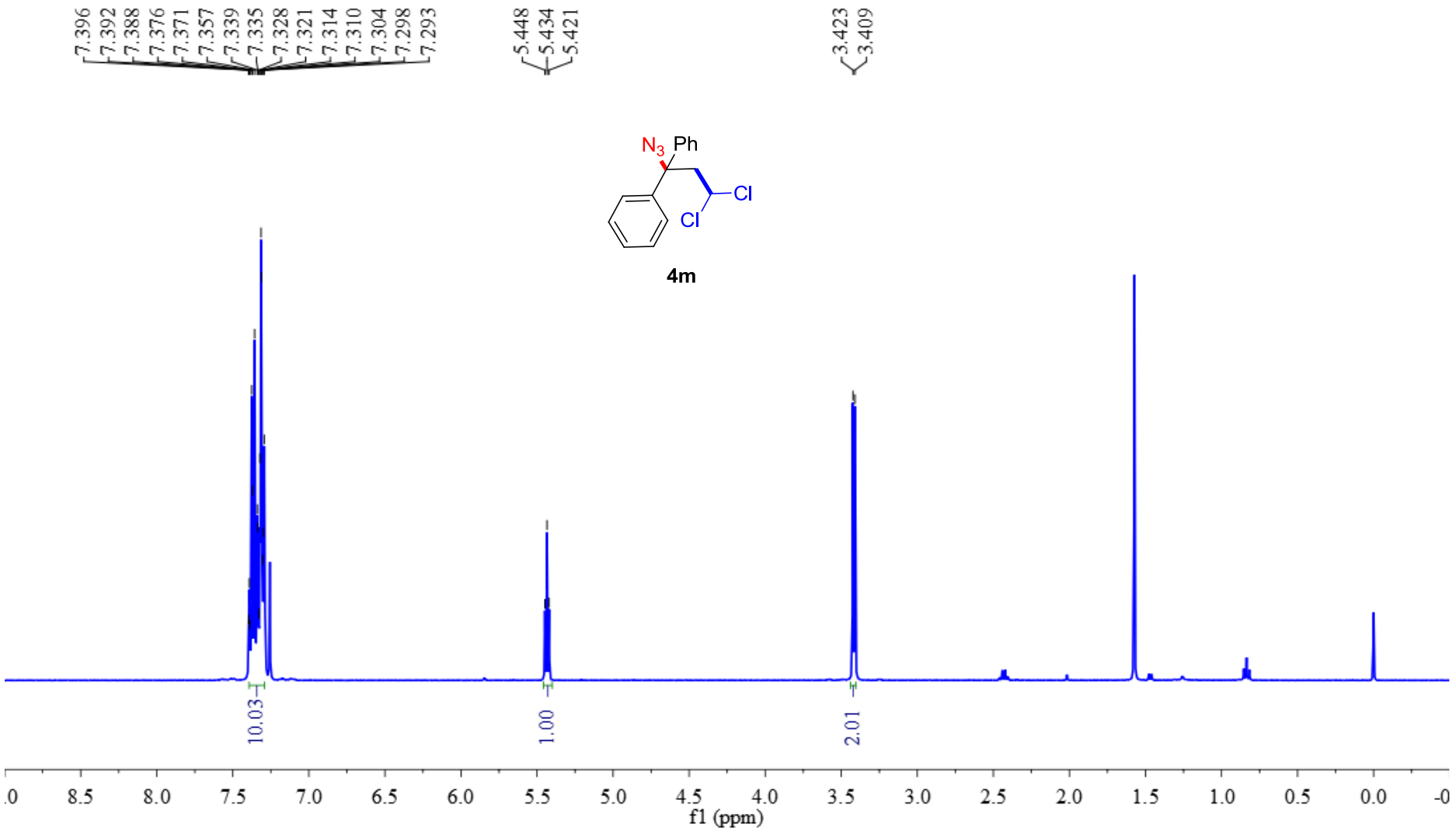

7.534
7.529
7.525
7.517
7.512
7.507
7.505
7.500
7.325
7.306
7.289
7.287
7.282
5.773
5.761
5.751
5.739
4.746
4.734
4.722
4.710
2.635
2.624
2.611
2.599
2.587
2.575
2.563
2.481
2.469
2.460
2.447
2.445
2.433
2.423
2.411
N3
Cl
Cl
Br
4k
1.99
2.01
1.00
1.01
1.04
1.02
f1 (ppm)

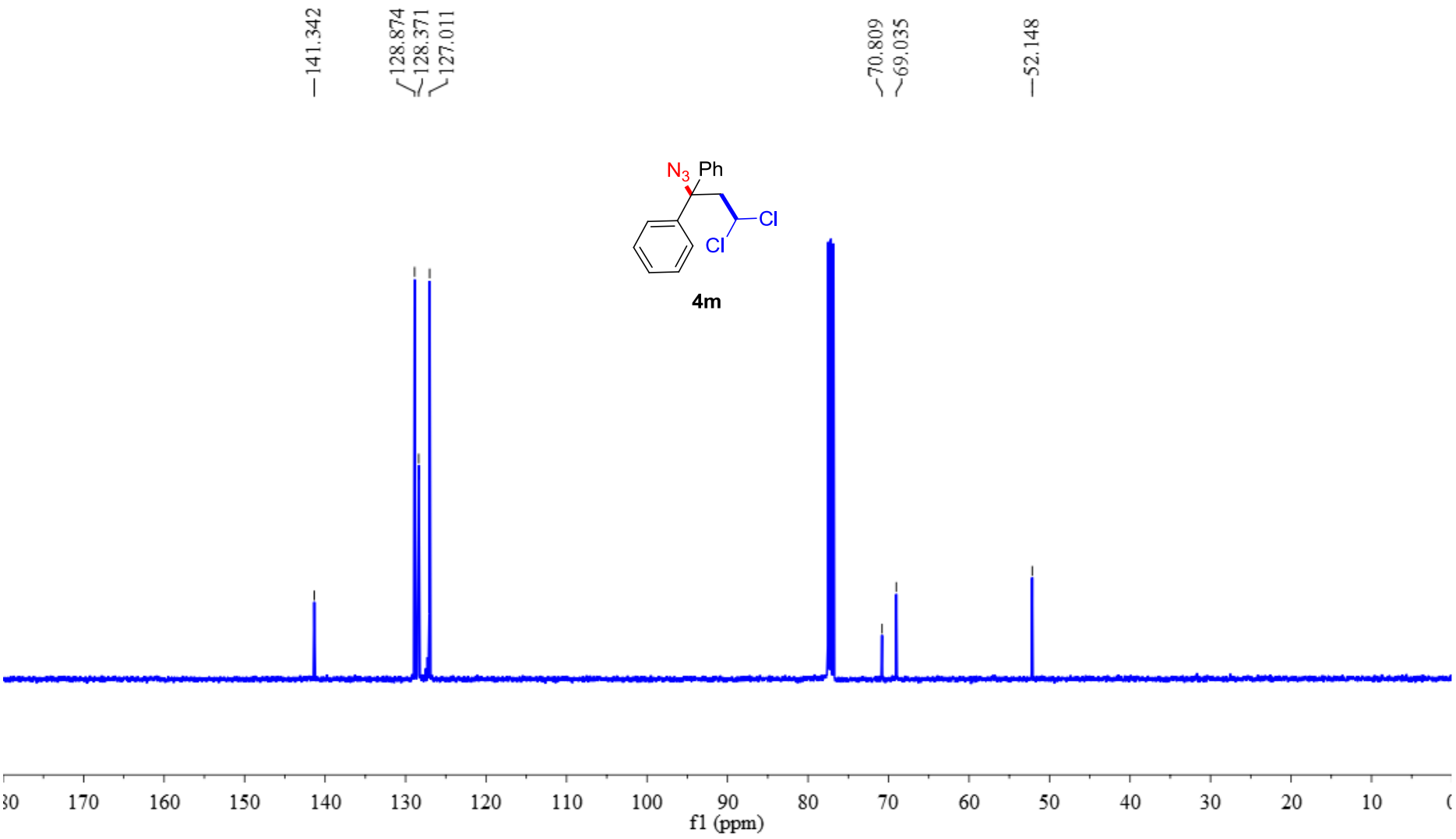

140.138
132.352
130.937
130.135
125.675
123.451
69.886
62.757
49.875
N3
Cl
Cl
Br
4k
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

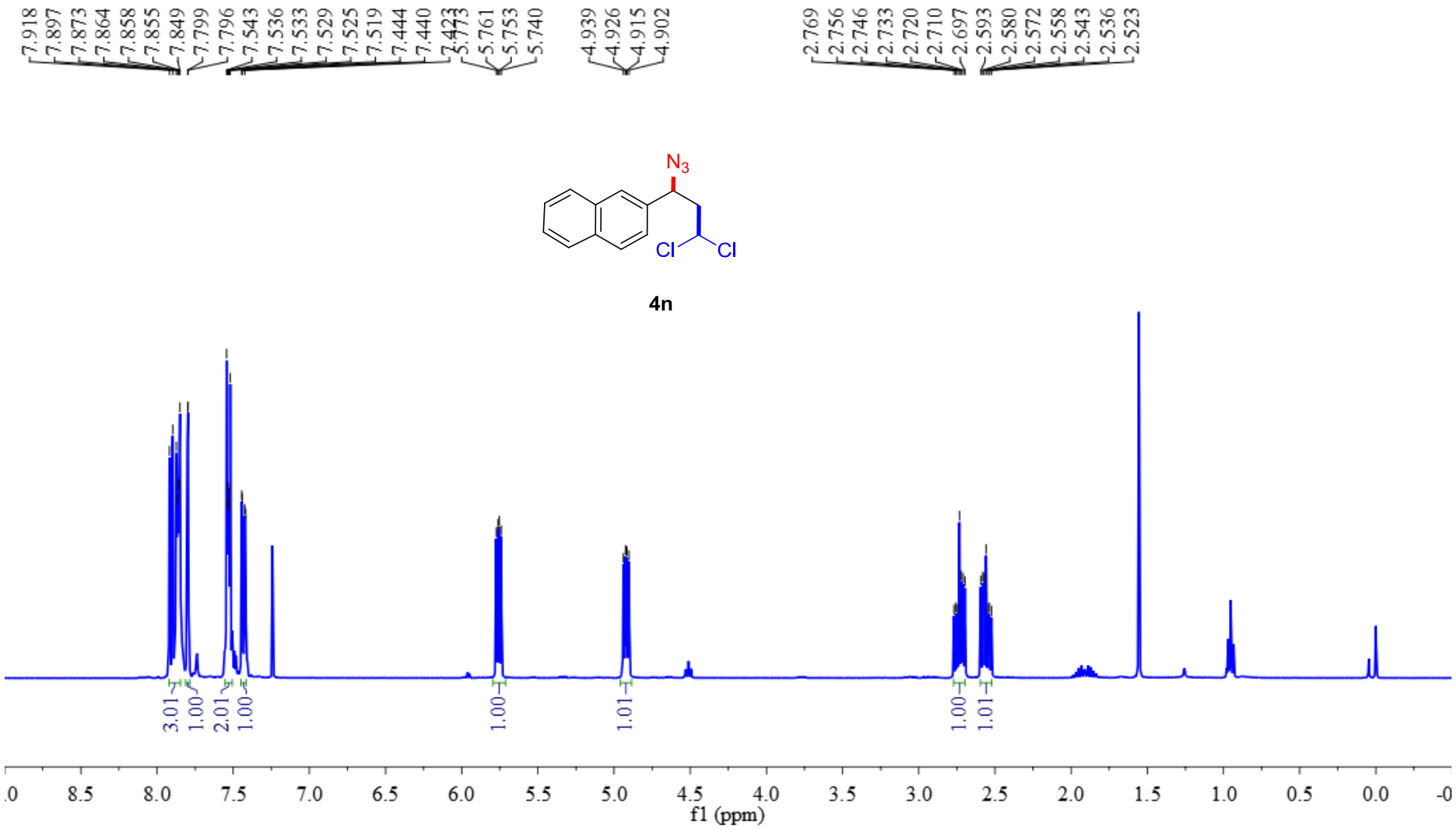

7.414
7.403
7.380
7.359
7.349
7.339
7.328
7.317
7.306
5.567
5.553
5.538
2.803
2.791
2.788
2.778
1.817
N3
Cl
Cl
4.02
1.04
1.00
2.01
3.01
f1 (ppm)

4l


141.668
129.086
128.215
125.587
68.972
65.464
55.385
25.837
N3
Cl
Cl
4l
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


7.396
7.392
7.388
7.376
7.371
7.357
7.339
7.335
7.328
7.321
7.314
7.310
7.304
7.298
7.293
5.448
5.434
5.421
3.423
3.409
N3
Ph
Cl
Cl
4m
10.03
1.00
2.01
f1 (ppm)


141.342
128.874
128.371
127.011
70.809
69.035
52.148
N3 Ph
Cl
Cl
4m
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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7.897
7.873
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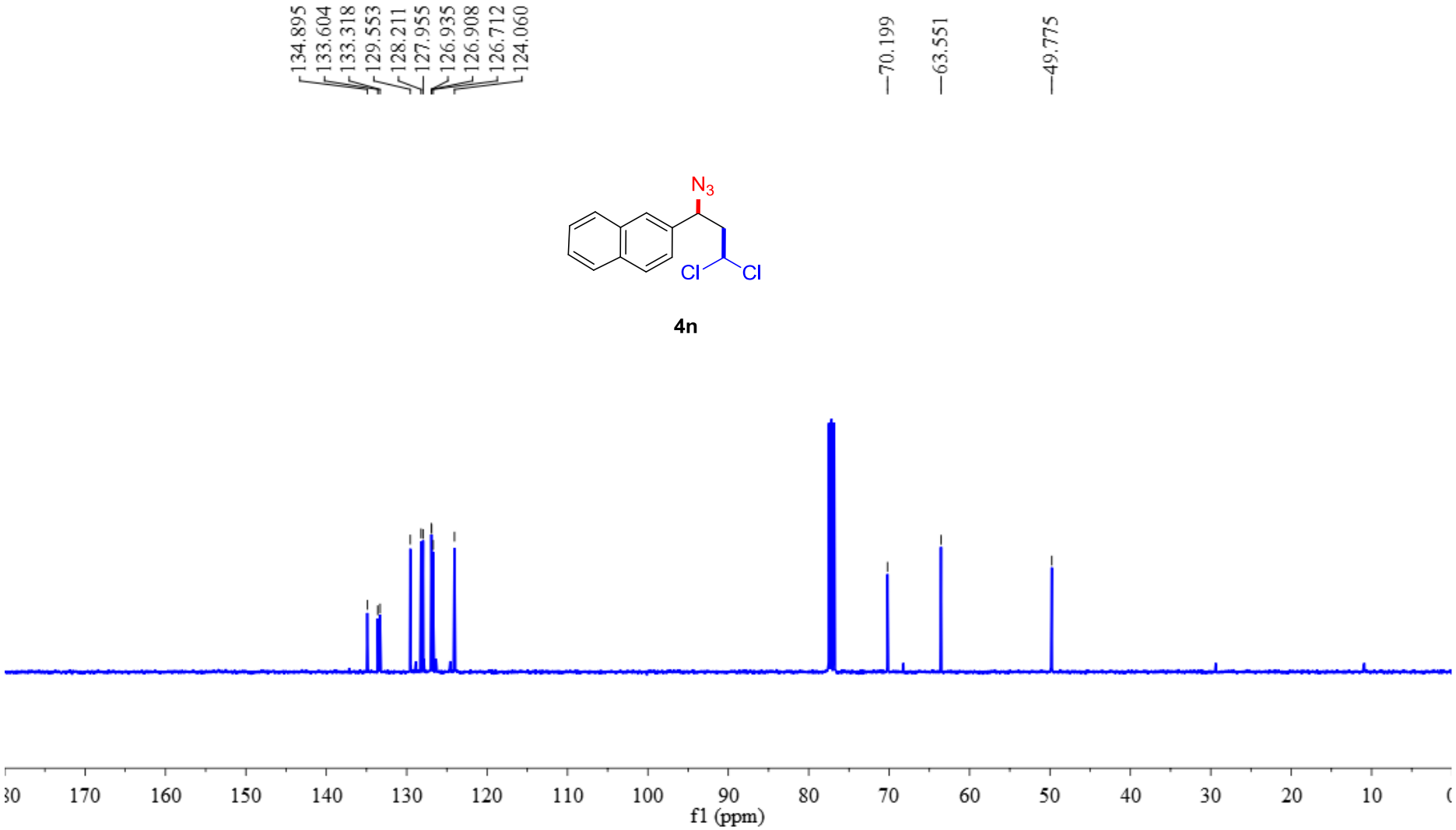
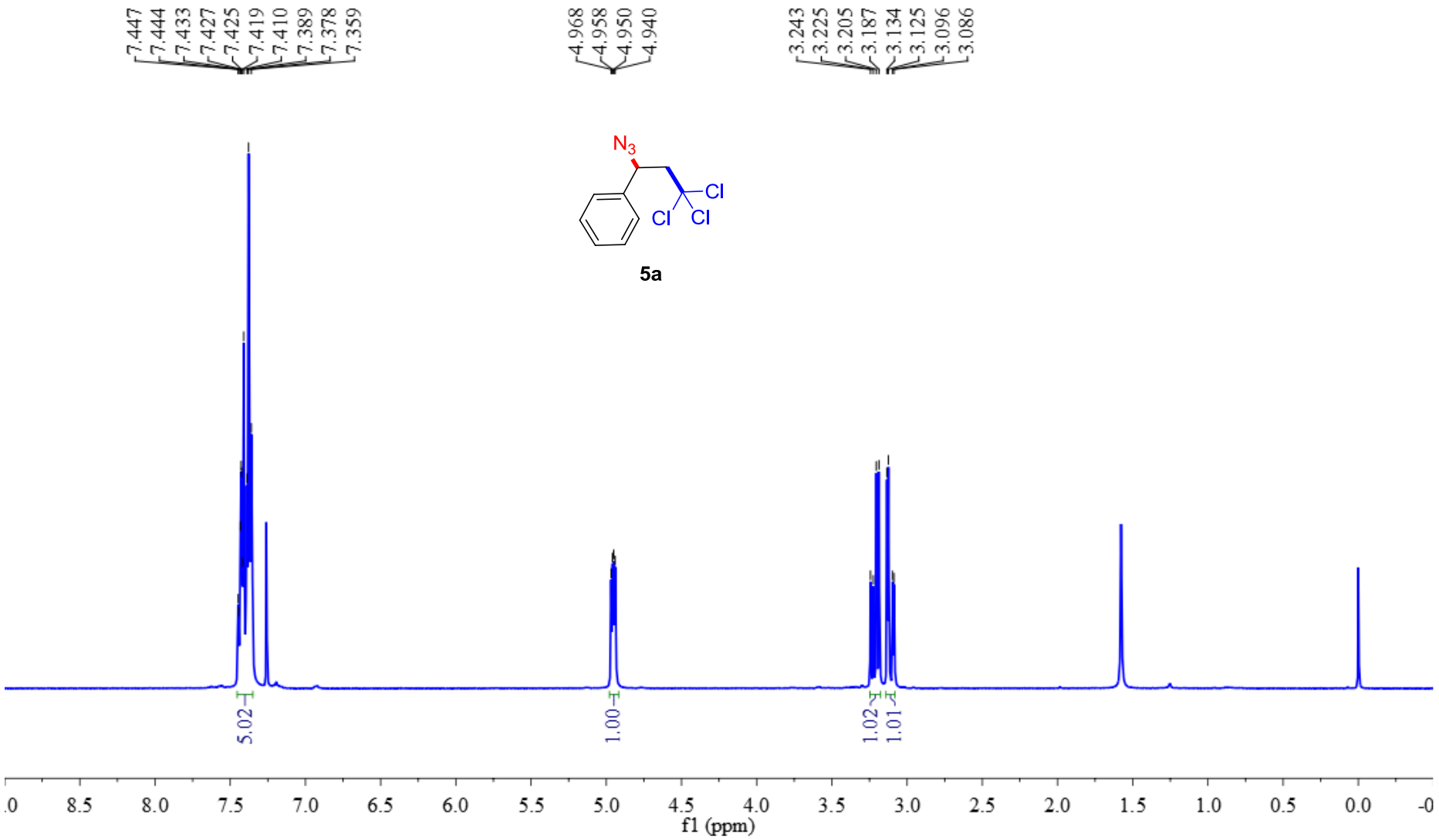
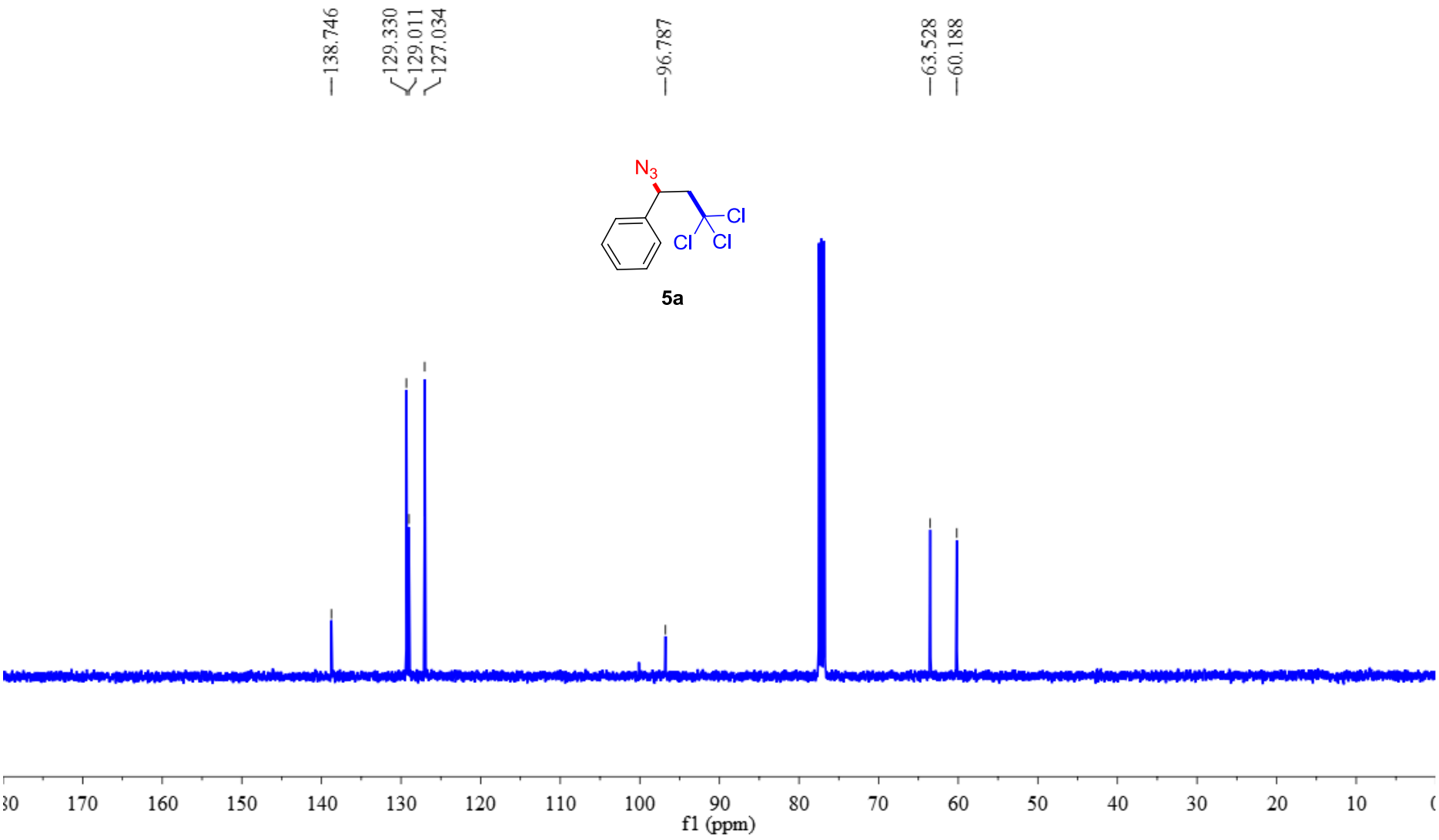
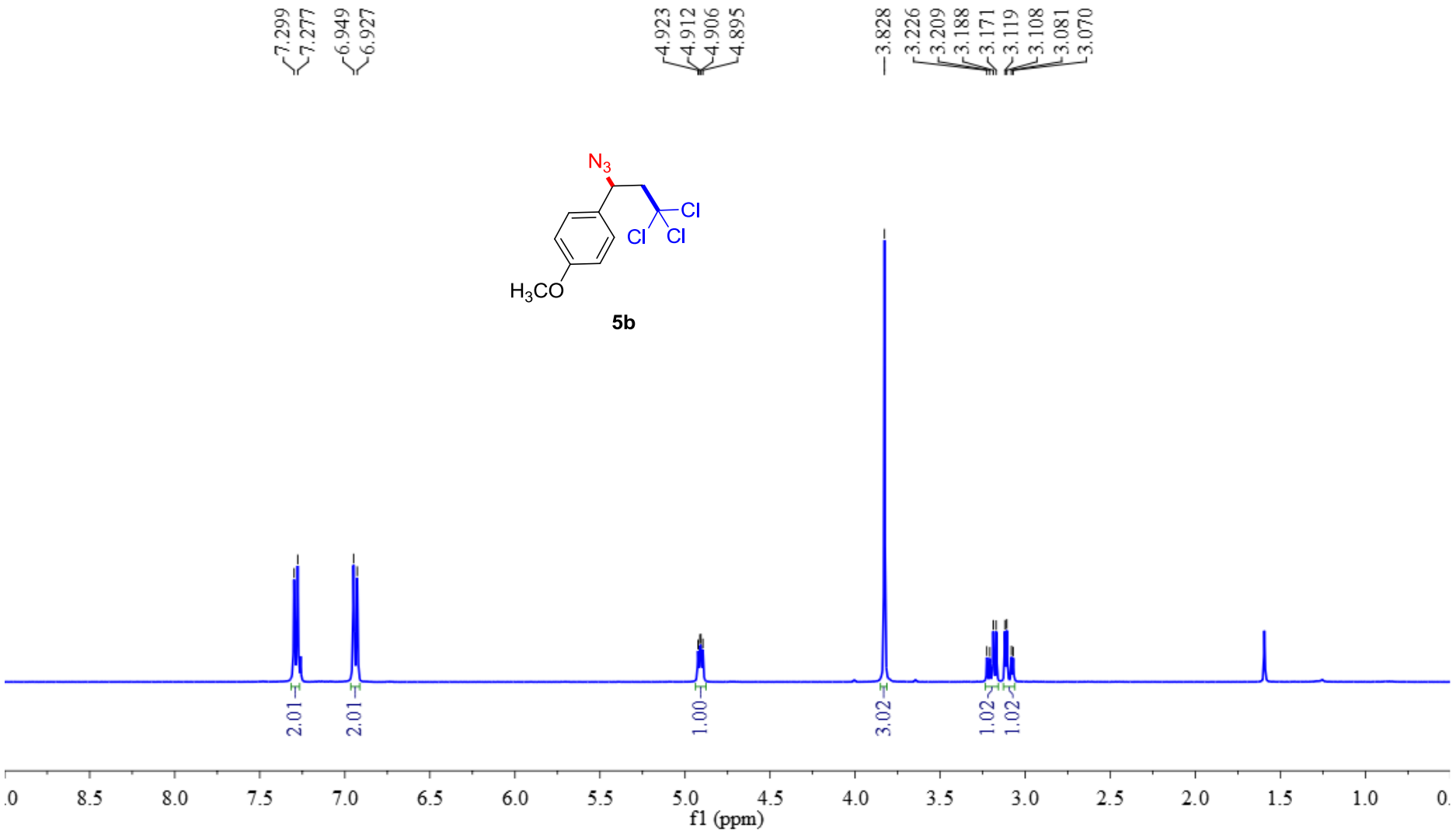
7.858
7.855
7.849
7.799
7.796
7.543
7.536
7.533
7.529
7.525
7.519
7.444
7.440
7.423
5.773
5.761
5.753
5.740
4.939
4.926
4.915
4.902
2.769
2.756
2.746
2.733
2.720
2.710
2.697
2.593
2.580
2.572
2.558
2.543
2.536
2.523
N3
Cl
Cl
4n
3.01
1.00
2.01
1.00
1.00
1.01
1.00
1.01
f1 (ppm)

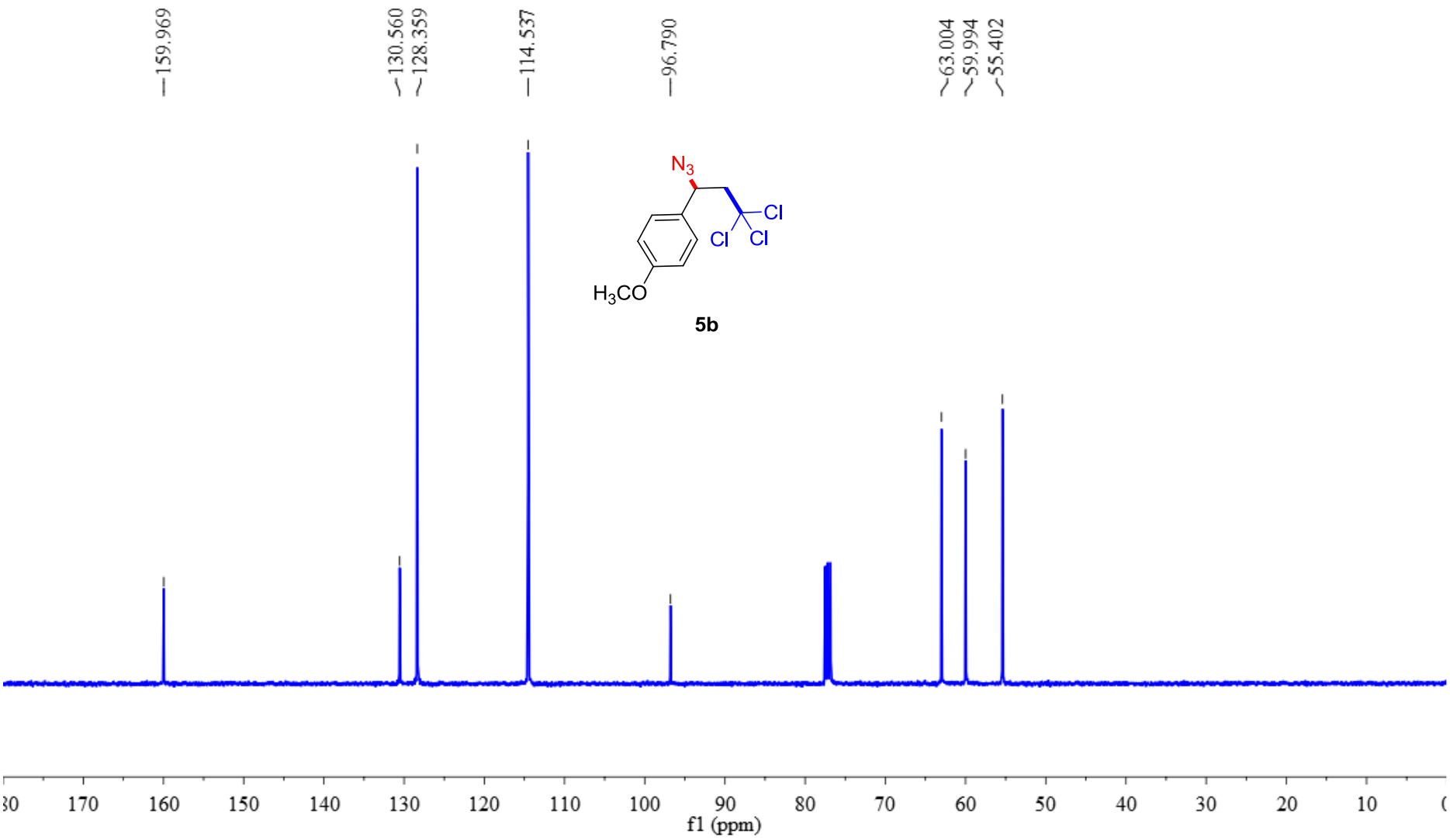

134.895
133.604
133.318
129.553
128.211
127.955
126.935
126.908
126.712
124.060
70.199
63.551
49.775
N3
Cl
Cl
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

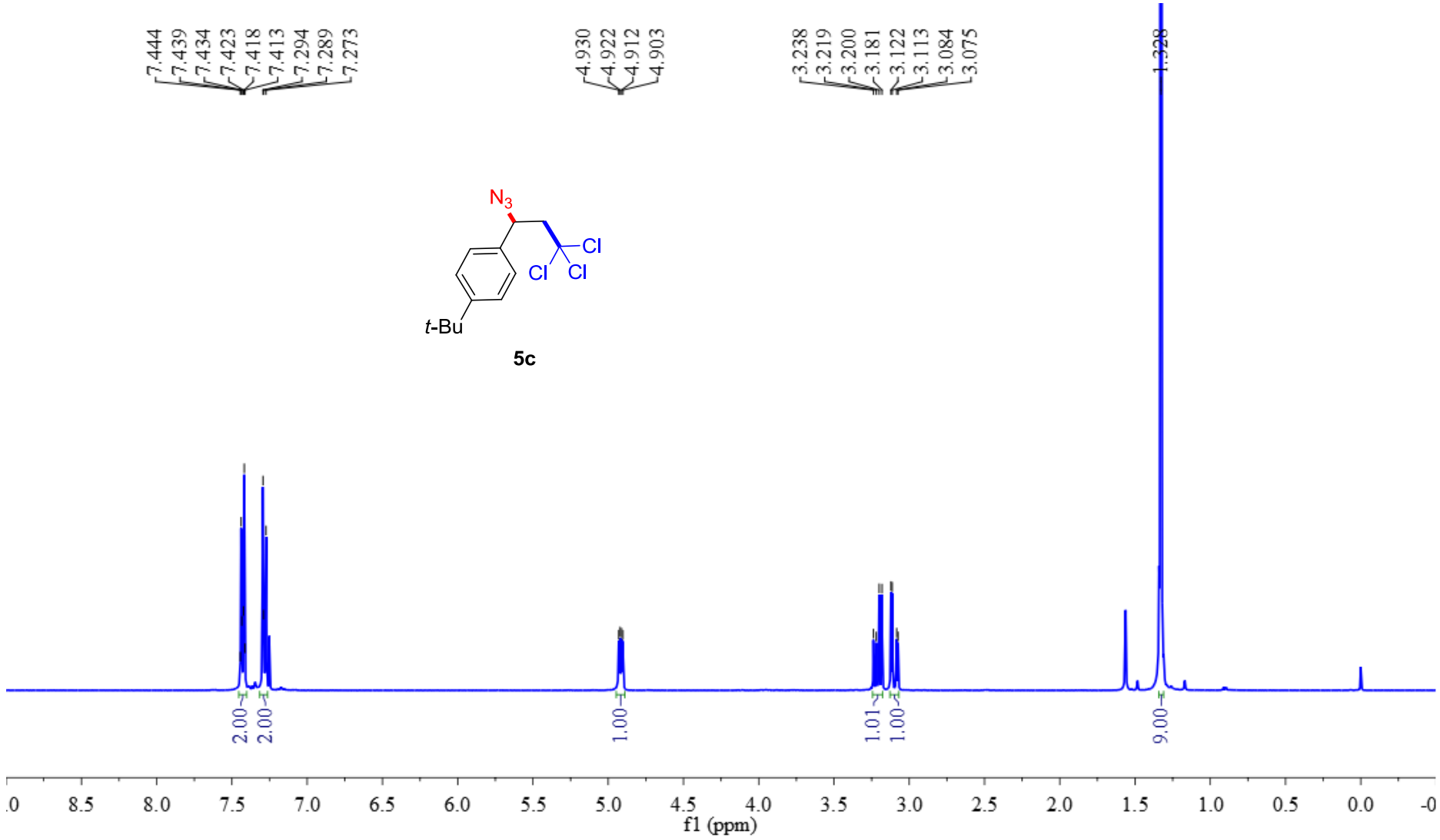
4n


7.447
7.444
7.433
7.427
7.425
7.419
7.410
7.389
7.378
7.359
4.968
4.958
4.950
4.940
3.243
3.225
3.205
3.187
3.134
3.125
3.096
3.086
N3
Cl
Cl
Cl
5a
5.02
1.00
1.02
1.01
f1 (ppm)

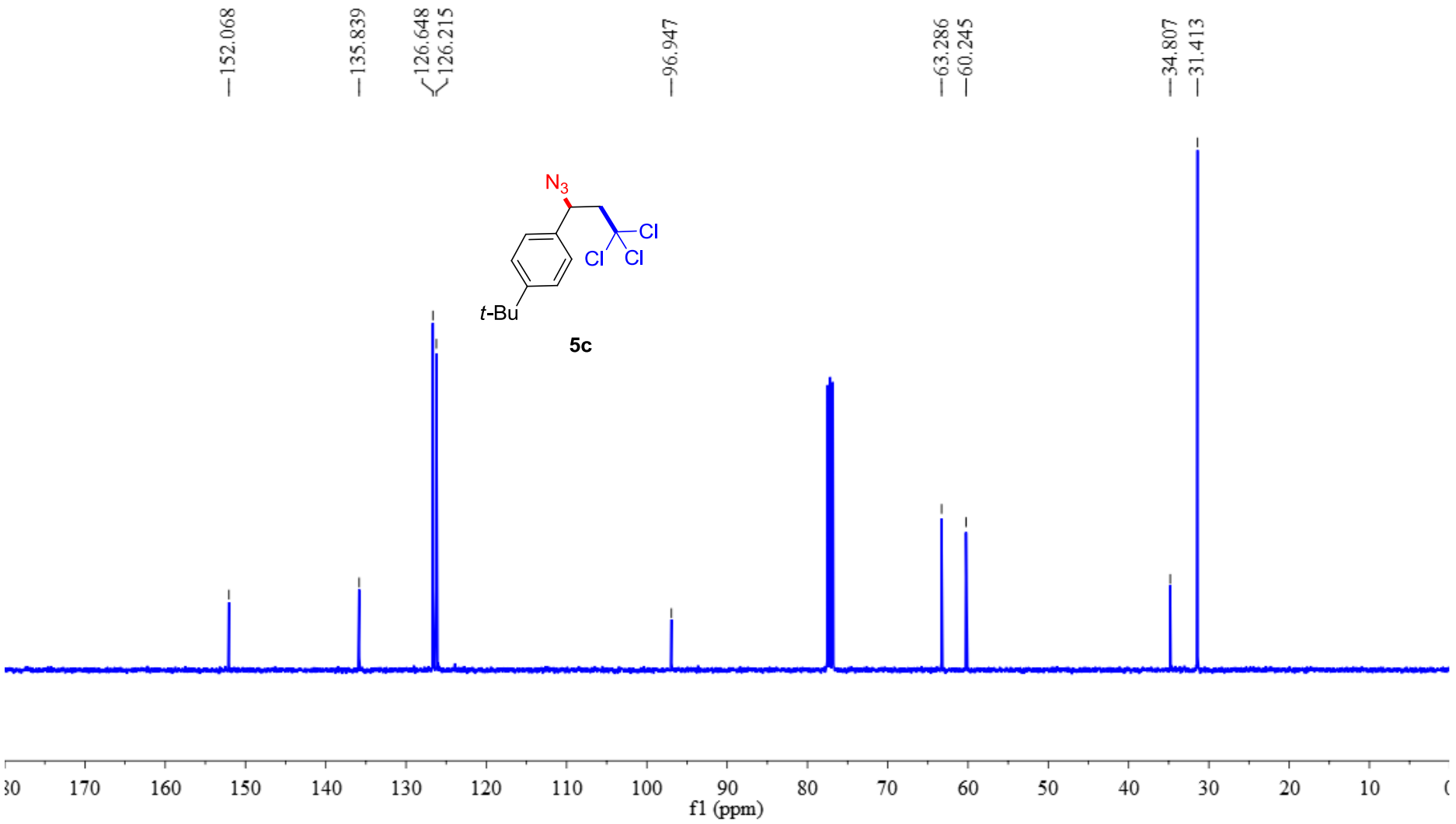

138.746
129.330
129.011
127.034
96.787
63.528
60.188
N3
Cl
Cl
Cl
5a
f1 (ppm)

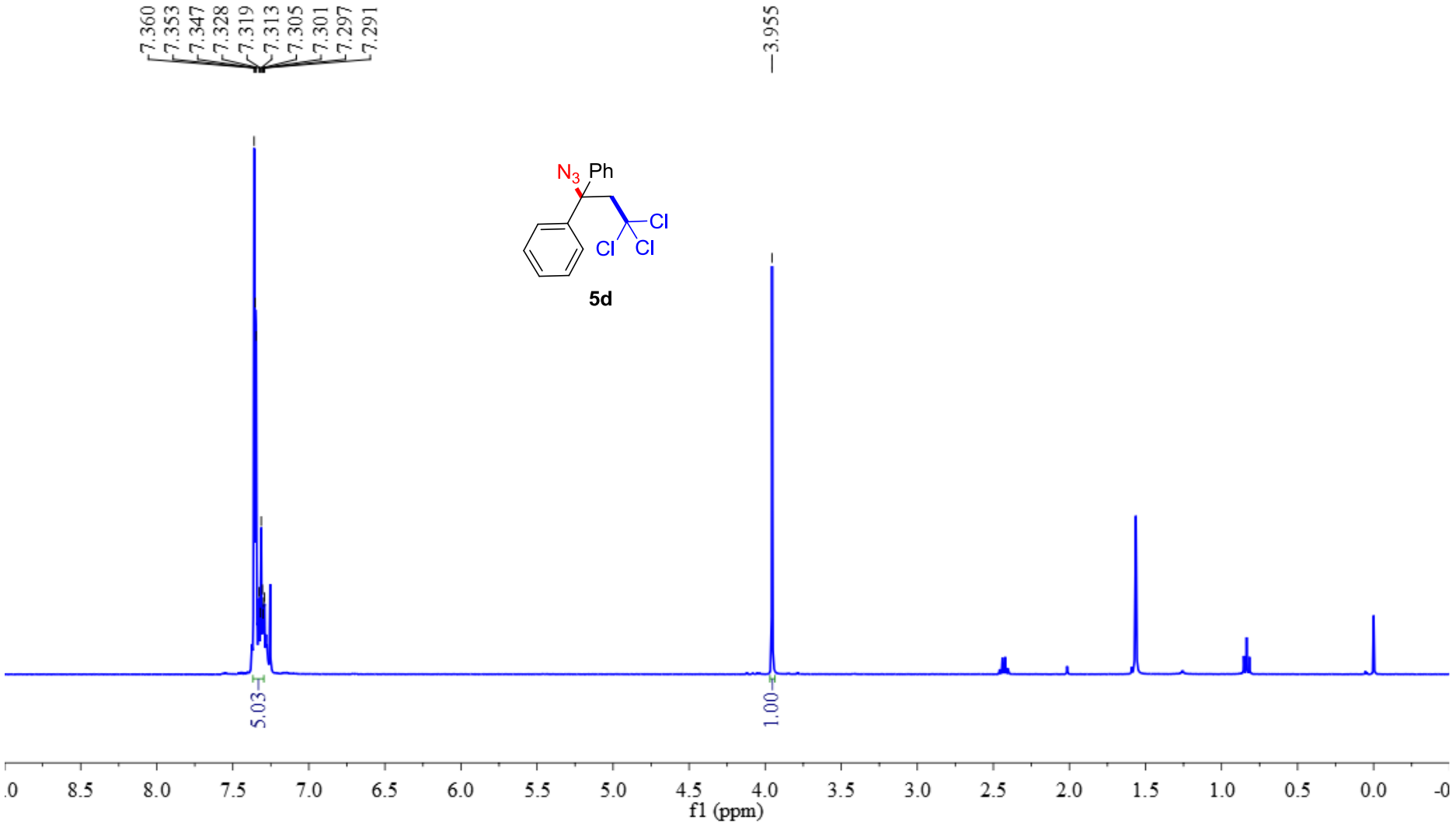

7.299
7.277
6.949
6.927
4.923
4.912
4.906
4.895
3.828
3.226
3.209
3.188
3.171
3.119
3.108
3.081
3.070
N3
Cl
Cl
Cl
H3CO
5b
2.01
2.01
1.00
3.02
1.02
1.02
f1 (ppm)

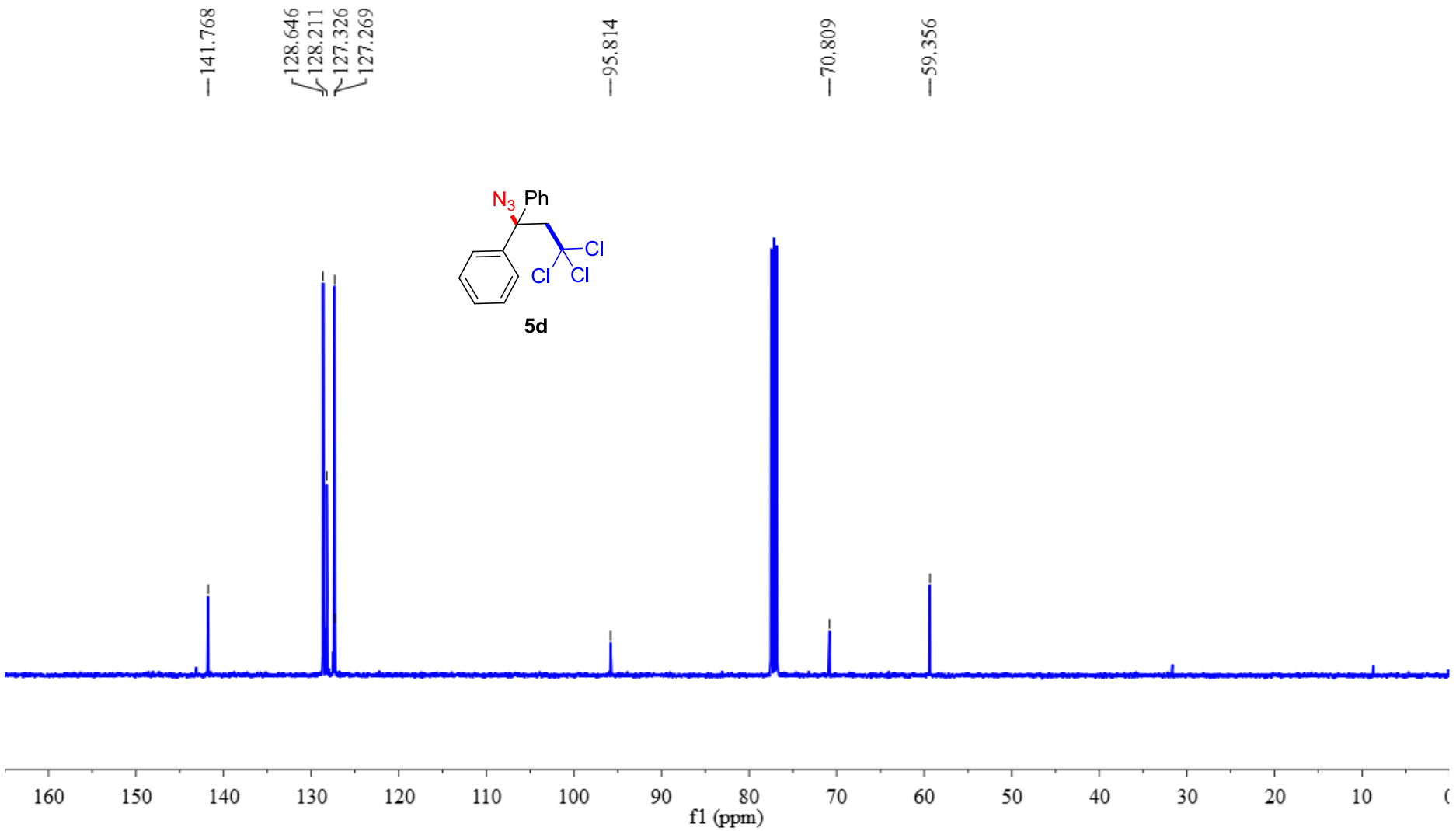

159.969
130.560
128.359
114.537
96.790
63.004
59.994
55.402
N3
Cl
Cl
Cl
H3CO
5b
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

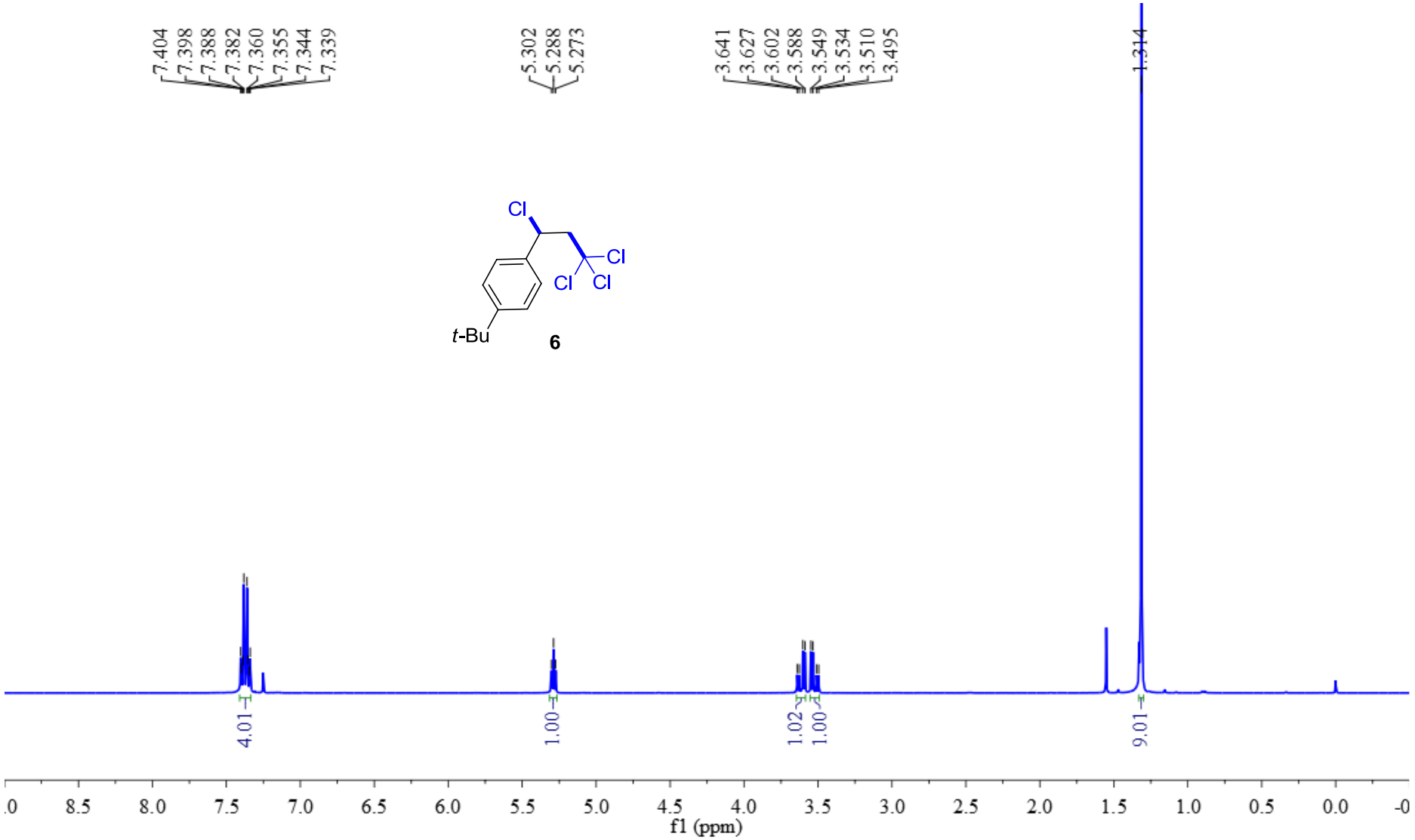

7.444
7.439
7.434
7.423
7.418
7.413
7.294
7.289
7.273
4.930
4.922
4.912
4.903
3.238
3.219
3.200
3.181
3.122
3.113
3.084
3.075
1.328
N3
Cl
Cl
Cl
t-Bu
2.00
2.00
1.00
1.01
1.00
9.00
f1 (ppm)

5c


152.068
135.839
126.648
126.215
96.947
63.286
60.245
34.807
31.413
N3
Cl
Cl
Cl
t-Bu
5c
f1 (ppm)


7.360
7.353
7.347
7.328
7.319
7.313
7.305
7.301
7.297
7.291
3.955
N3 Ph
Cl
Cl
Cl
5d
5.03
1.00
f1 (ppm)


141.768
128.646
128.211
127.326
127.269
95.814
70.809
59.356
N3 Ph
Cl
Cl
Cl
5d
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


7.404
7.398
7.388
7.382
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7.355
7.344
7.339
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5.288
5.273
3.641
3.627
3.602
3.588
3.549
3.534
3.510
3.495
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Cl
Cl
Cl
Cl
t-Bu
6
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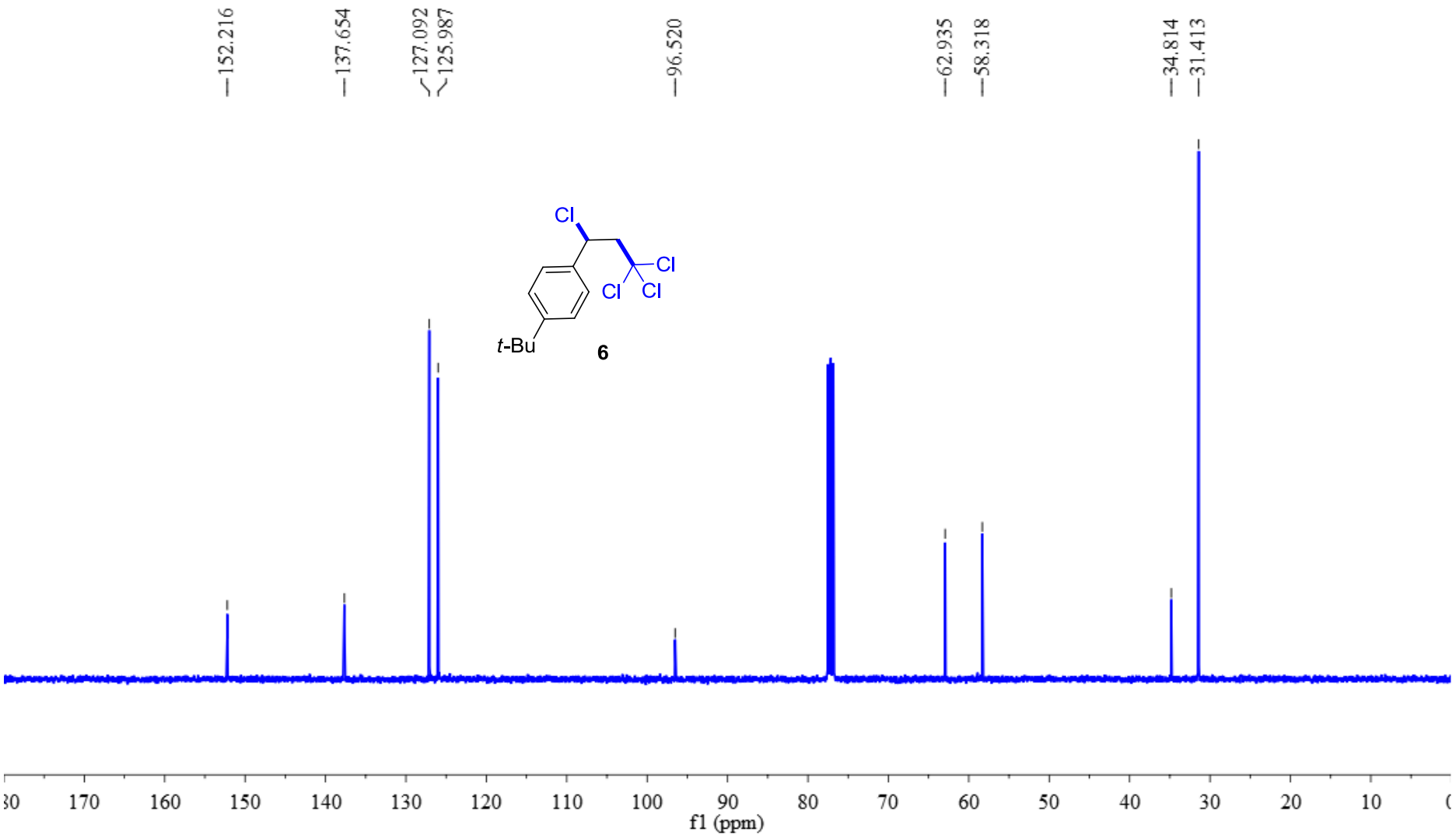
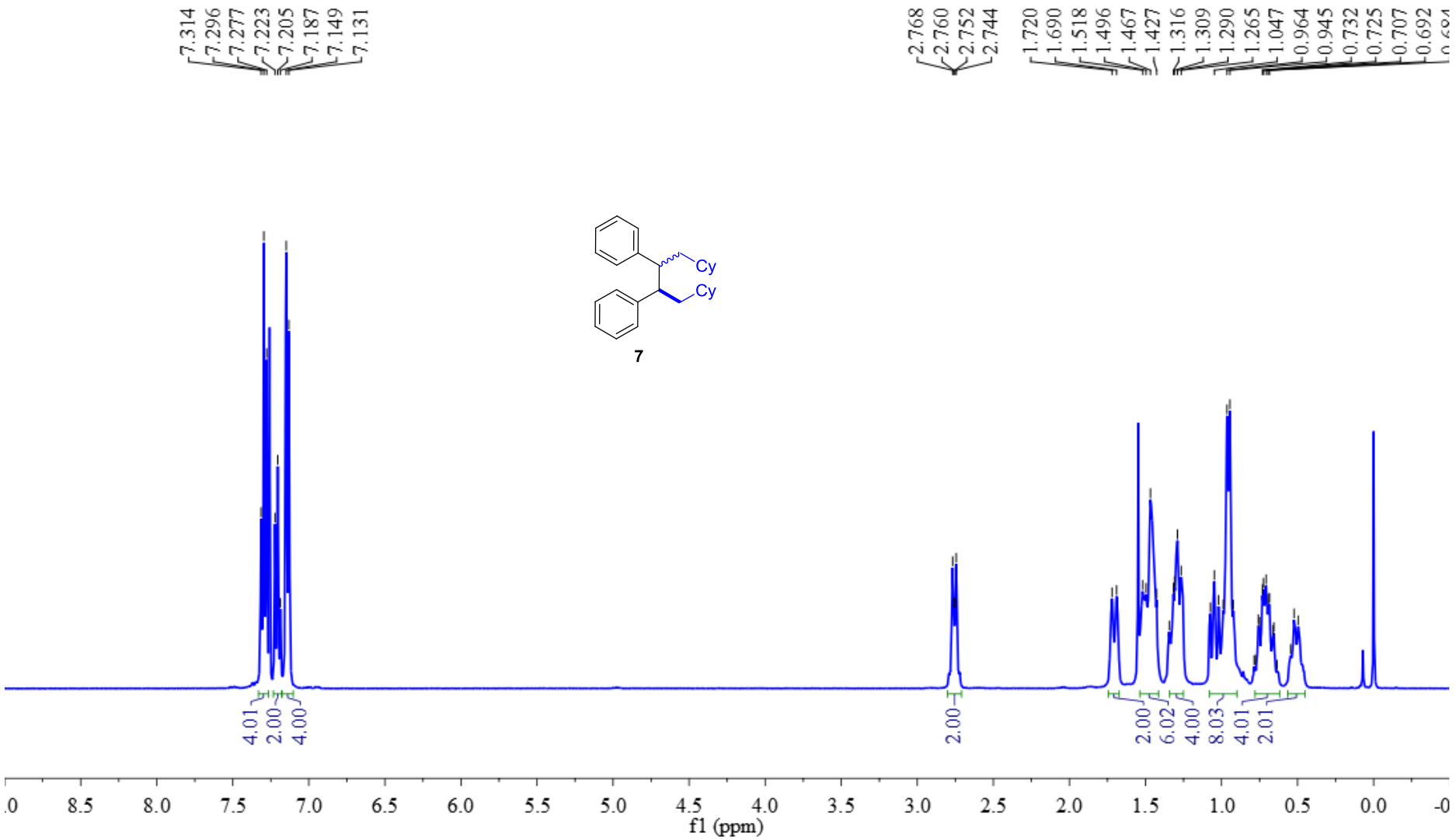
1.00
1.02
1.00
9.01
f1 (ppm)


152.216
137.654
127.092
125.987
96.520
62.935
58.318
34.814
31.413
Cl
Cl
Cl
Cl
t-Bu
6
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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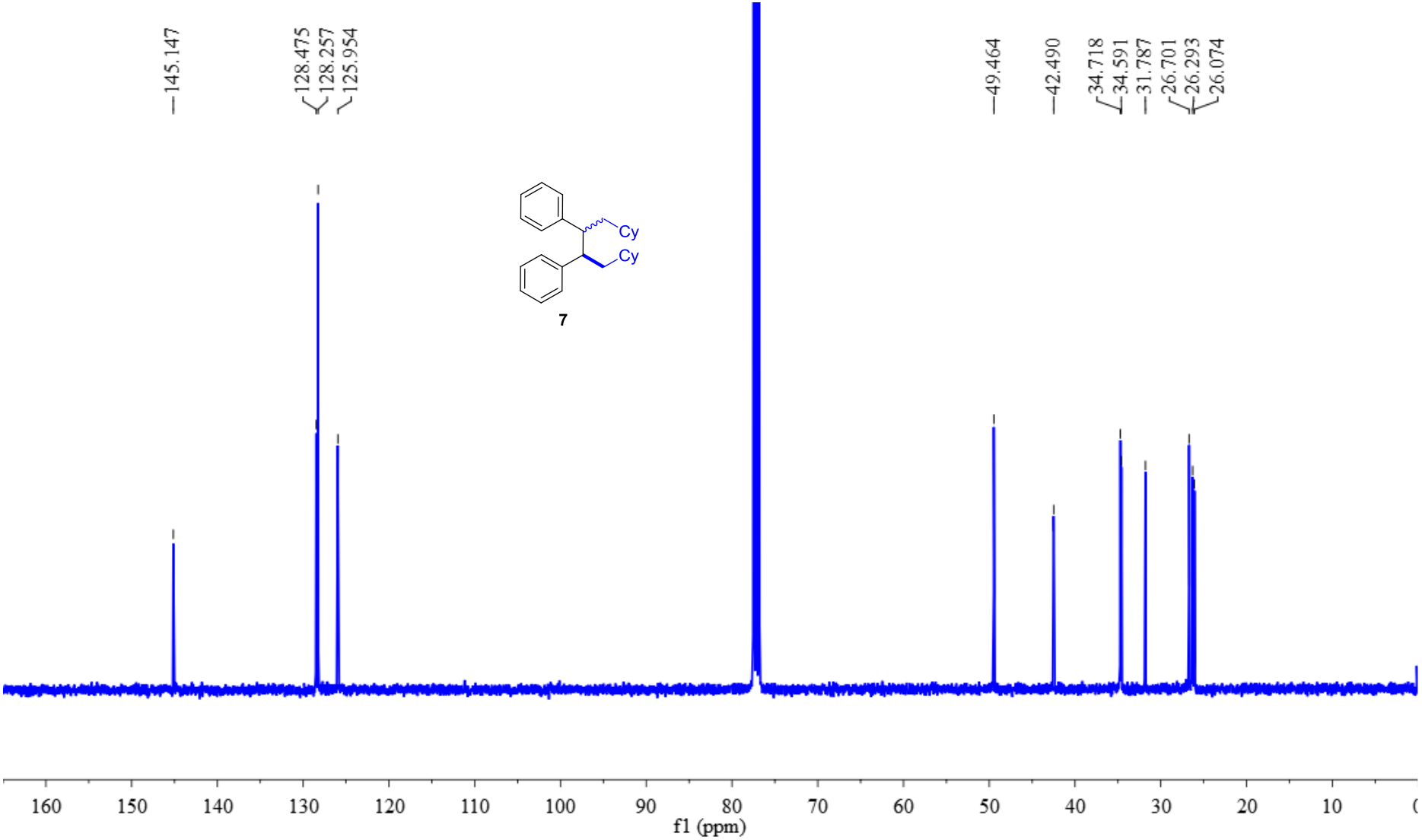
7.277
7.223
7.205
7.187
7.149
7.131
2.768
2.760
2.752
2.744
1.720
1.690
1.518
1.496
1.467
1.427
1.316
1.309
1.290
1.265
1.047
0.964
0.945
0.732
0.725
0.707
0.692
Cy
Cy
7
4.01
2.00
4.00
2.00
2.00
6.02
4.00
8.03
4.01
2.01
f1 (ppm)


145.147
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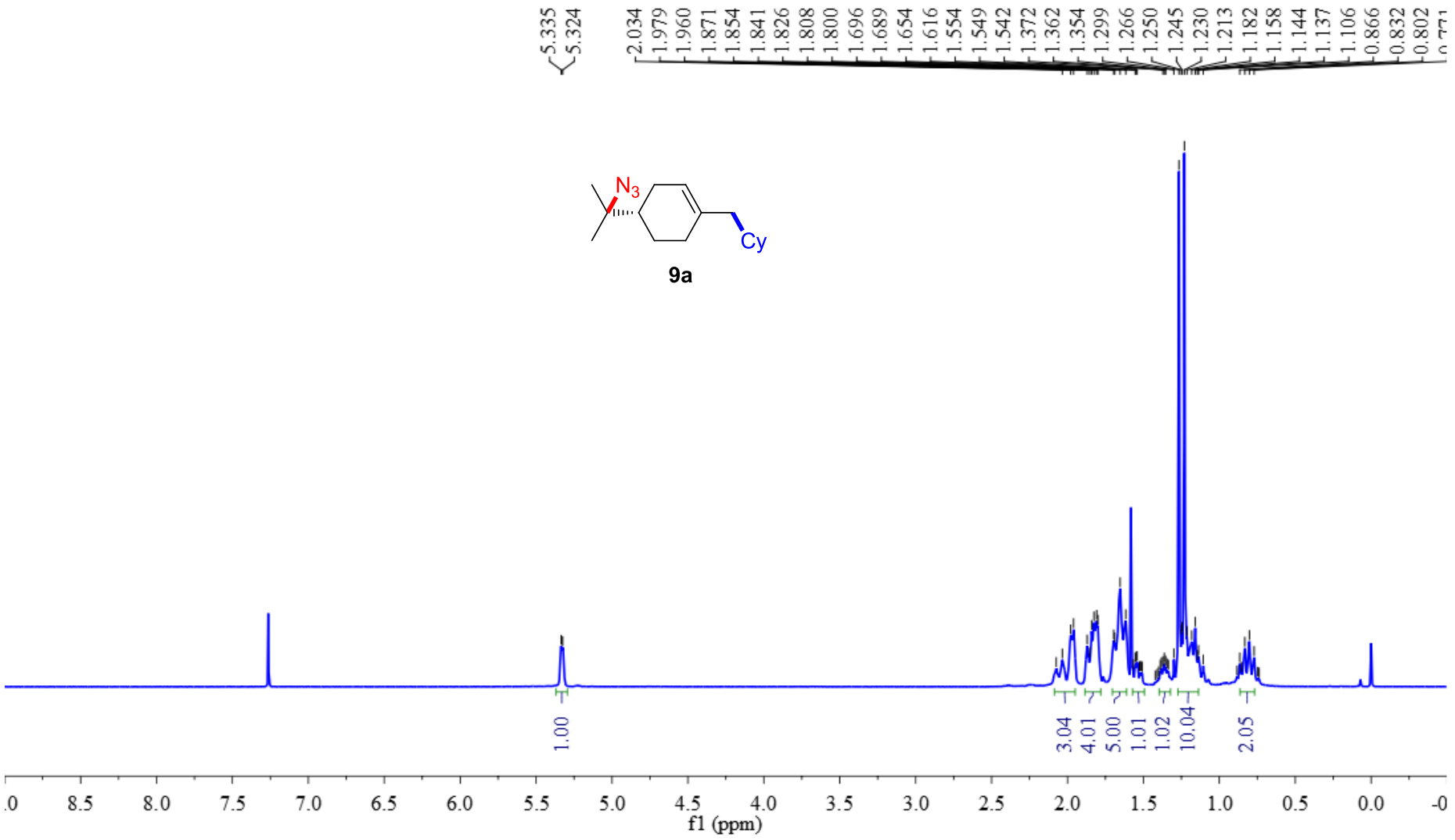
128.257
125.954
49.464
42.490
34.718
34.591
31.787
26.701
26.293
26.074
Cy
Cy
7
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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5.324
2.034
1.979
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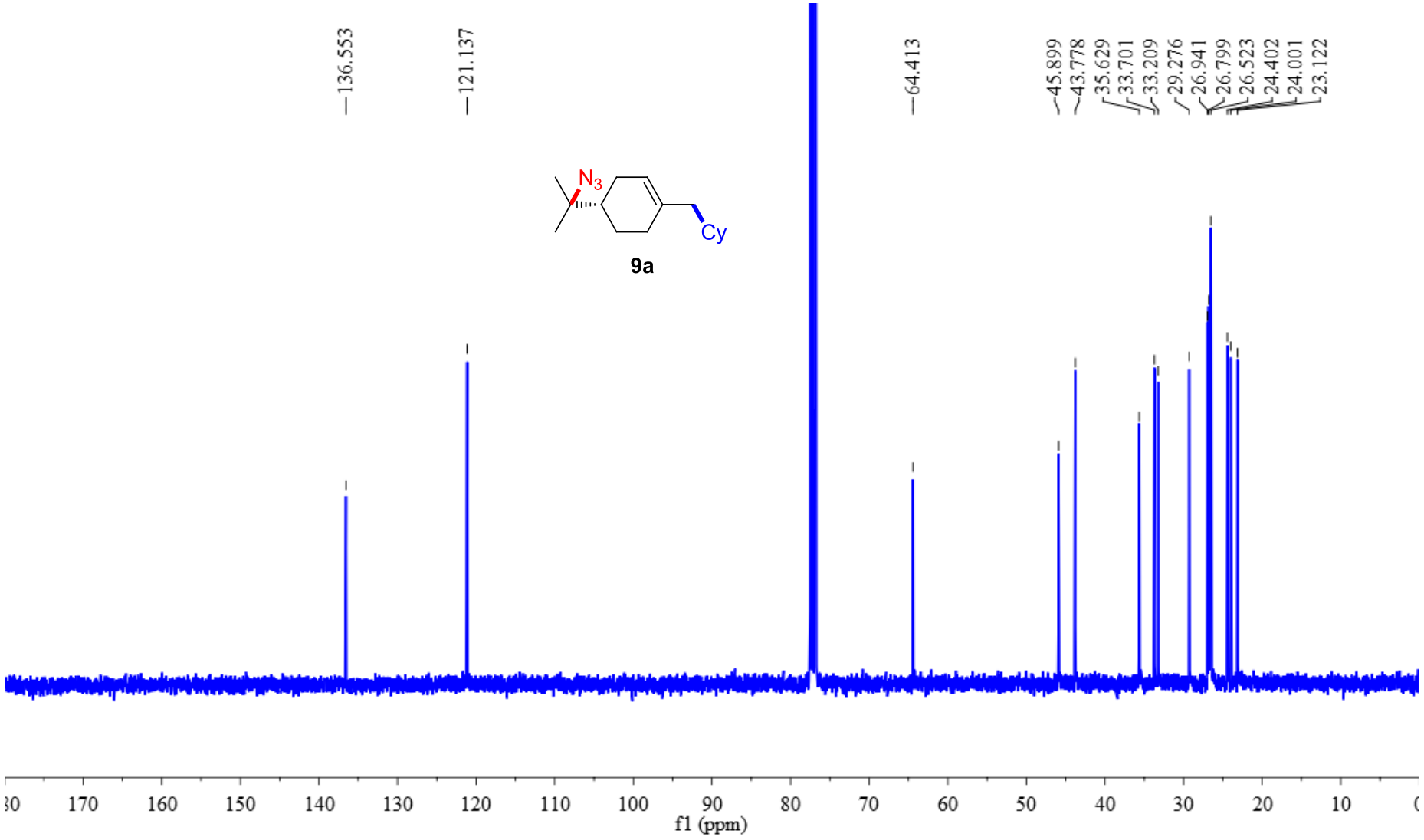
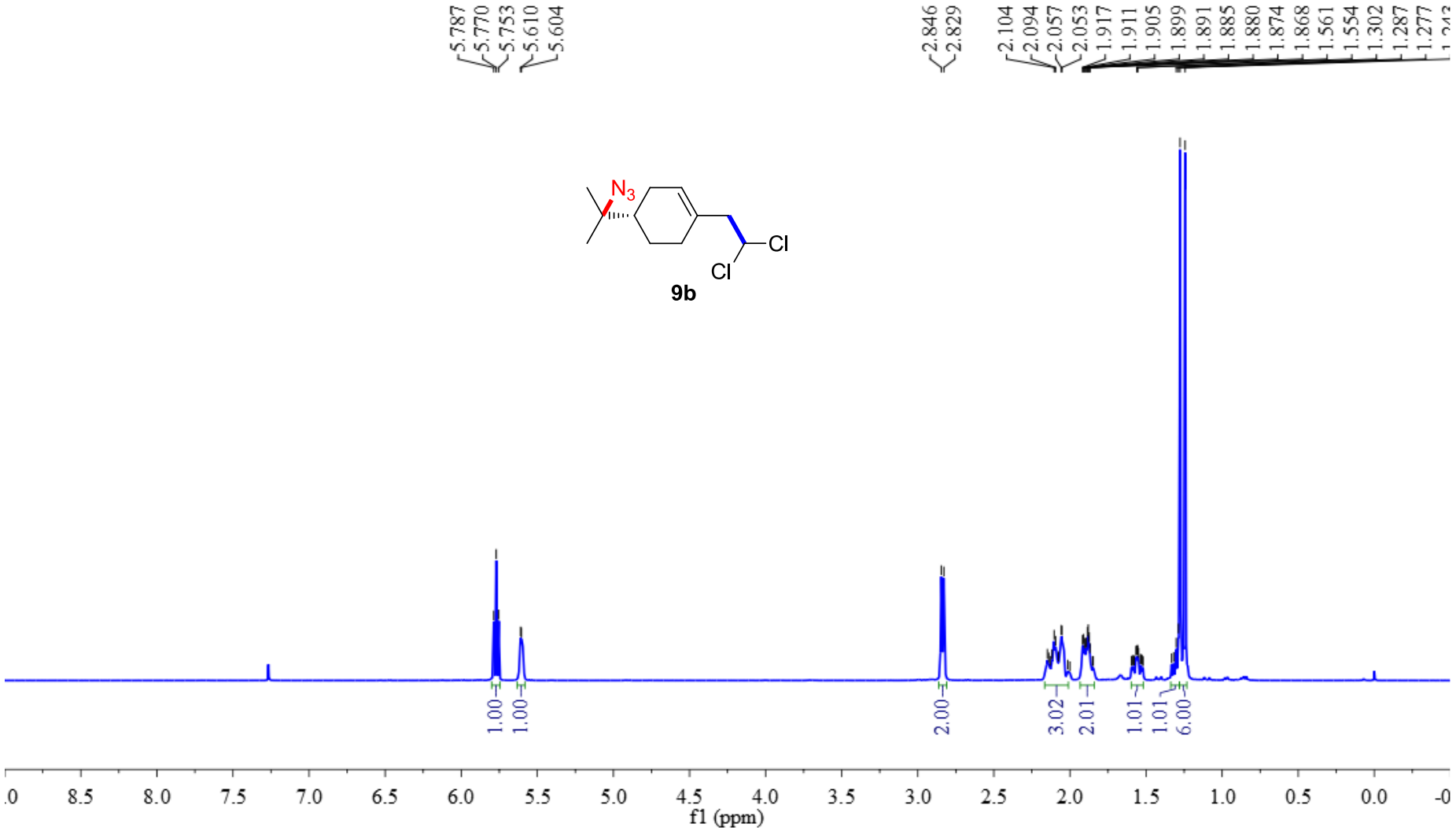
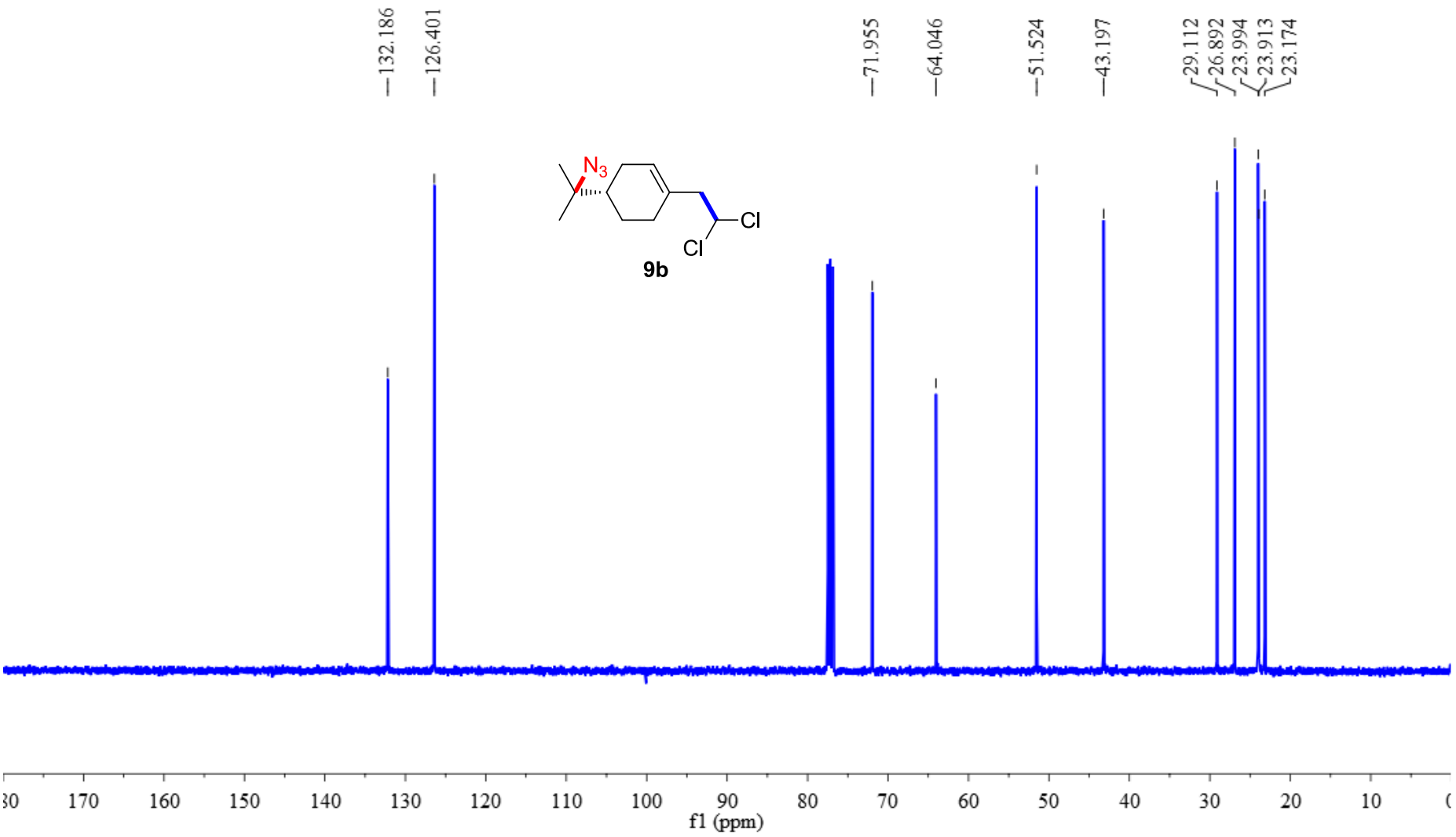
1.871
1.854
1.841
1.826
1.808
1.800
1.696
1.689
1.654
1.616
1.554
1.549
1.542
1.372
1.362
1.354
1.299
1.266
1.250
1.245
1.230
1.213
1.182
1.158
1.144
1.137
1.106
0.866
0.832
0.802
N3
Cy
9a
1.00
3.04
4.01
5.00
1.01
1.02
10.04
2.05
f1 (ppm)


136.553
121.137
64.413
45.899
43.778
35.629
33.701
33.209
29.276
26.941
26.799
26.523
24.402
24.001
23.122
N3
Cy
9a
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


5.787
5.770
5.753
5.610
5.604
2.846
2.829
2.104
2.094
2.057
2.053
1.917
1.911
1.905
1.899
1.891
1.885
1.880
1.874
1.868
1.561
1.554
1.302
1.287
1.277
N3
Cl
Cl
9b
1.00
1.00
2.00
3.02
2.01
1.01
1.01
6.00
f1 (ppm)


132.186
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51.524
43.197
29.112
26.892
23.994
23.913
23.174
N3
Cl
Cl
9b
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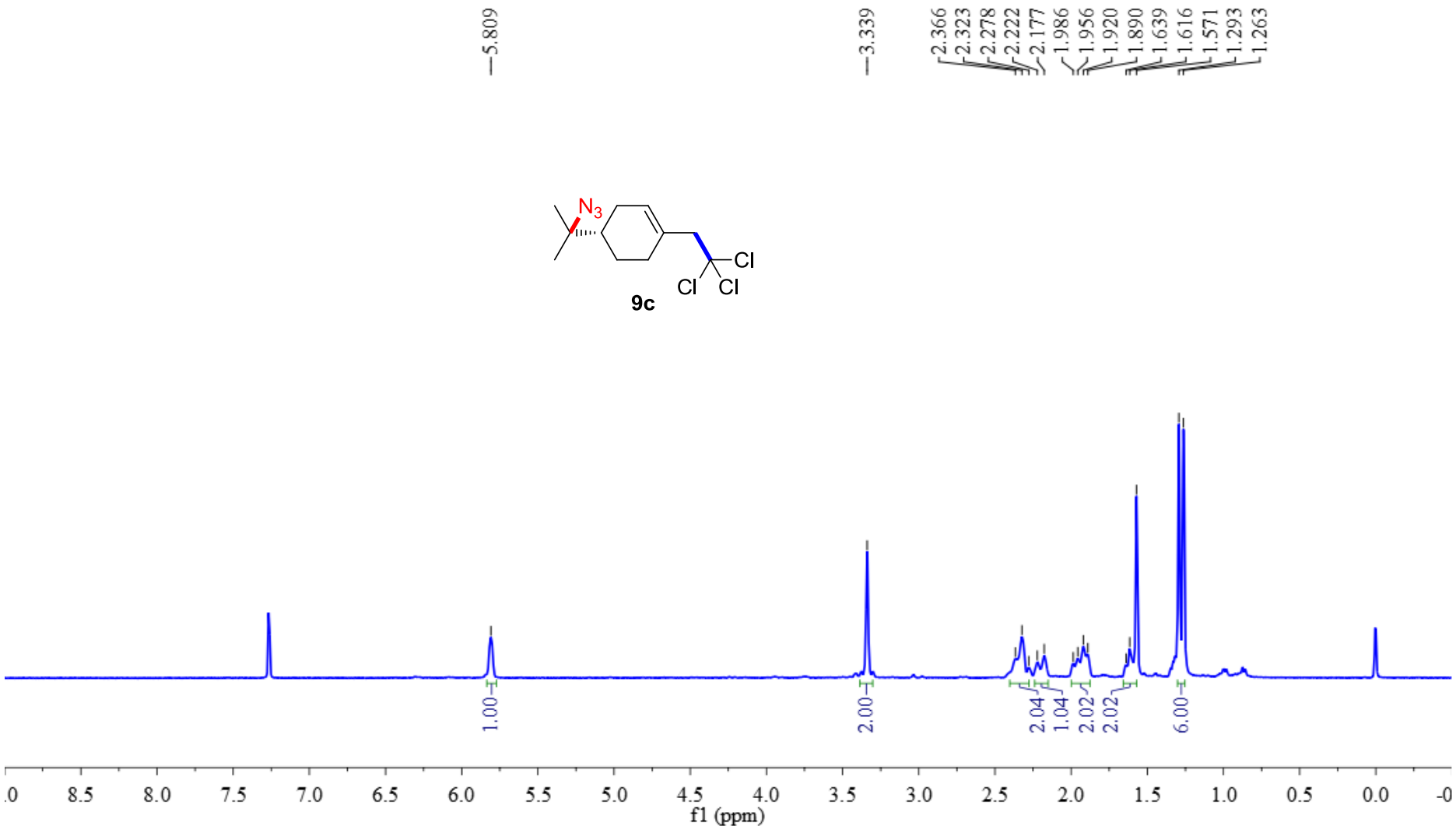
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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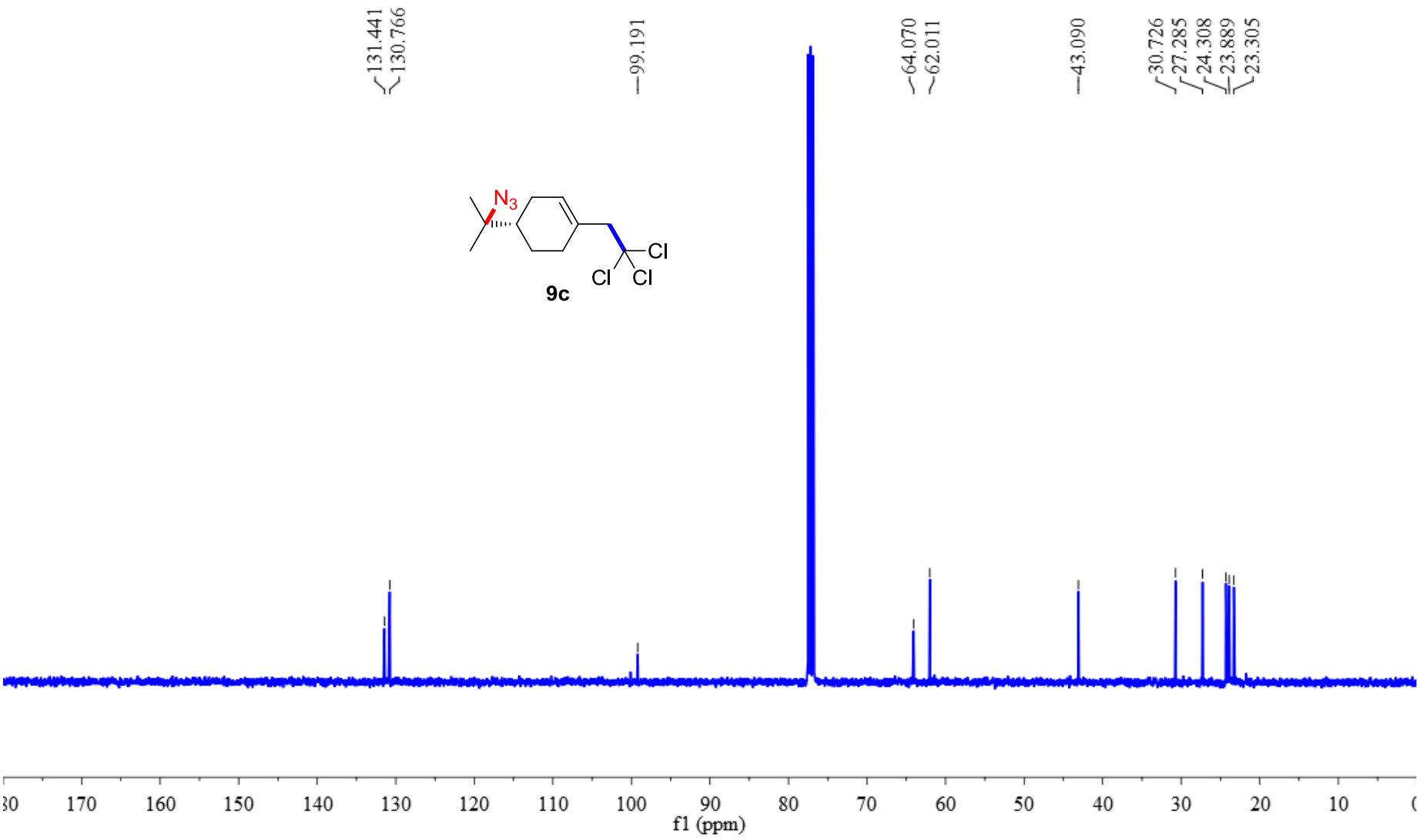
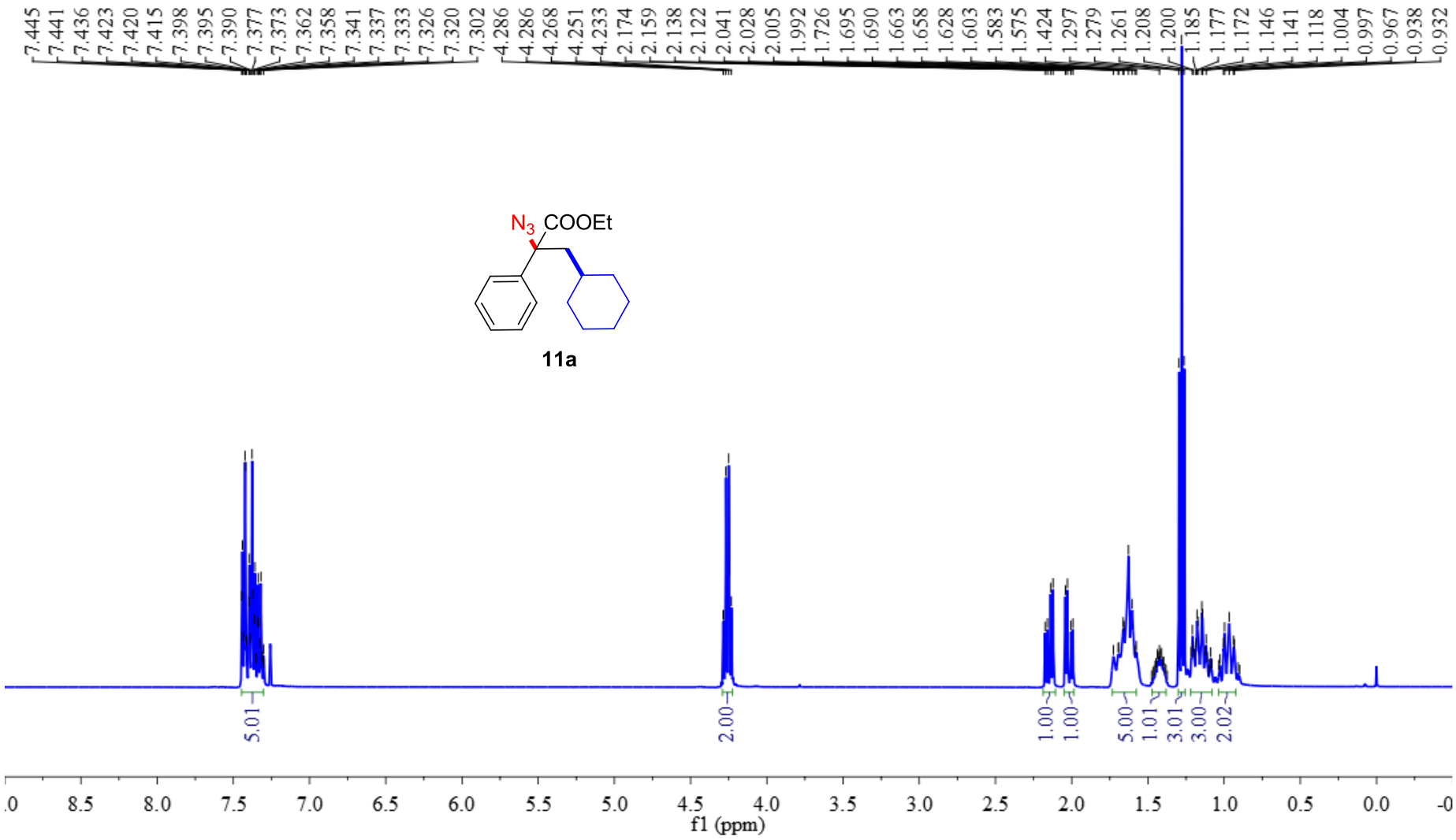
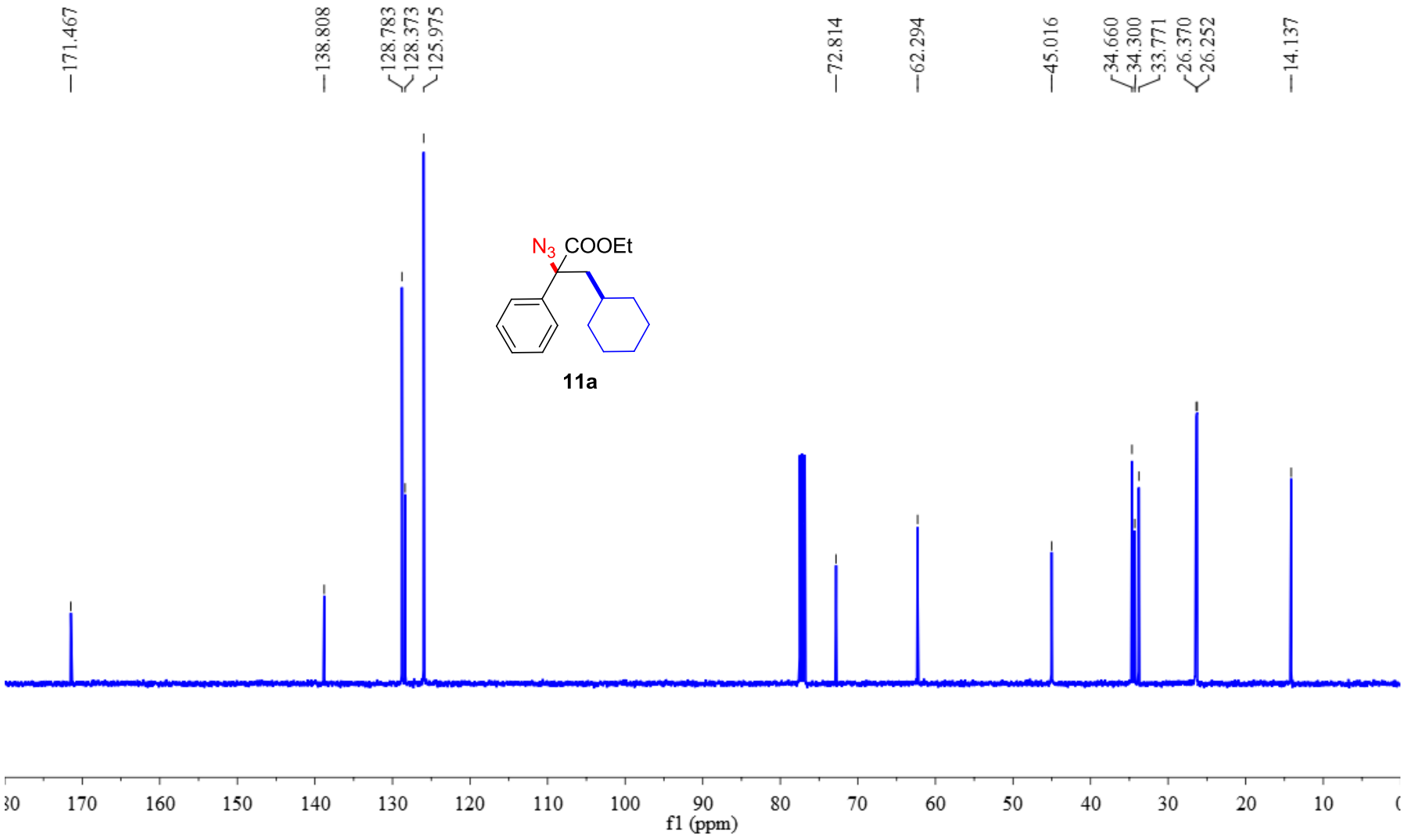
2.366
2.323
2.278
2.222
2.177
1.986
1.956
1.920
1.890
1.639
1.616
1.571
1.293
1.263
N3
Cl
Cl
Cl
9c
1.00
2.00
2.04
1.04
2.02
2.02
6.00
f1 (ppm)


131.441
130.766
99.191
64.070
62.011
43.090
30.726
27.285
24.308
23.889
23.305
N3
Cl
Cl
Cl
9c
f1 (ppm)


7.445
7.441
7.436
7.423
7.420
7.415
7.398
7.395
7.390
7.377
7.373
7.362
7.358
7.341
7.337
7.333
7.326
7.320
7.302
4.286
4.268
4.251
4.233
2.174
2.159
2.138
2.122
2.041
2.028
2.005
1.992
1.726
1.695
1.690
1.663
1.658
1.628
1.603
1.583
1.575
1.424
1.297
1.279
1.261
1.208
1.200
1.185
1.177
1.172
1.146
1.141
1.118
1.004
0.997
0.967
0.938
0.932
N3 COOEt
11a
5.01
2.00
1.00
1.00
5.00
1.01
3.01
3.00
2.02
f1 (ppm)


171.467
138.808
128.783
128.373
125.975
72.814
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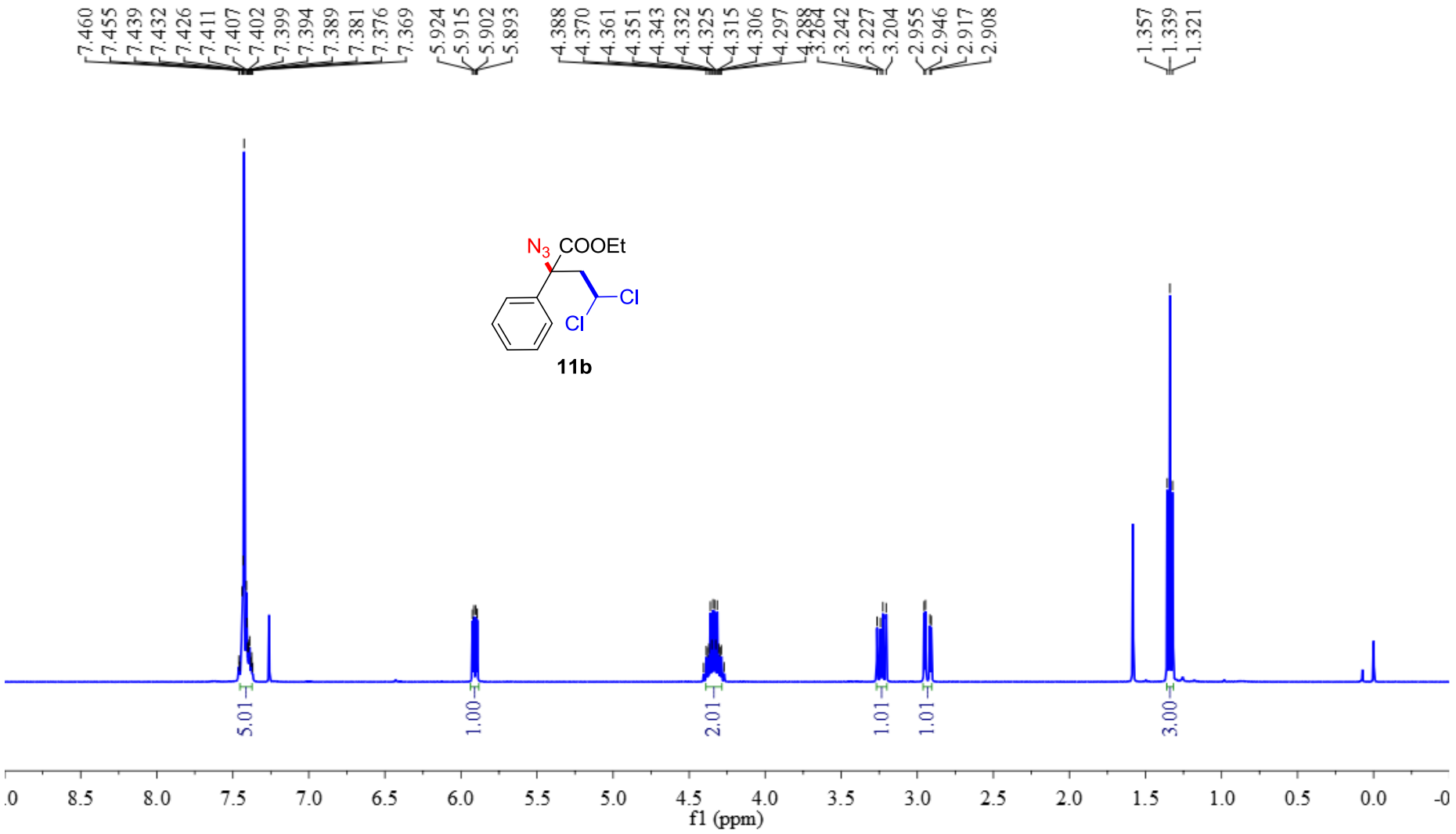
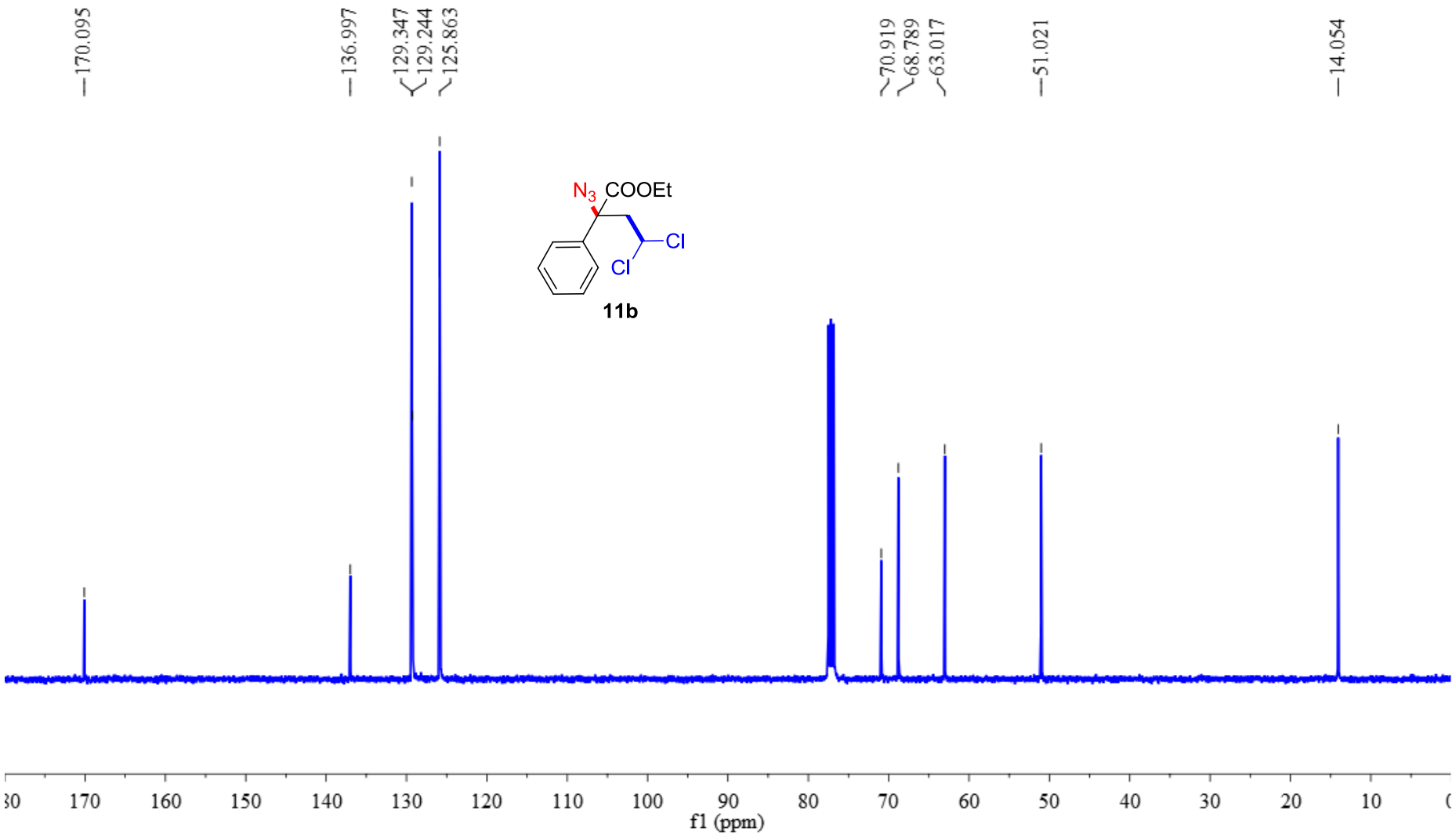
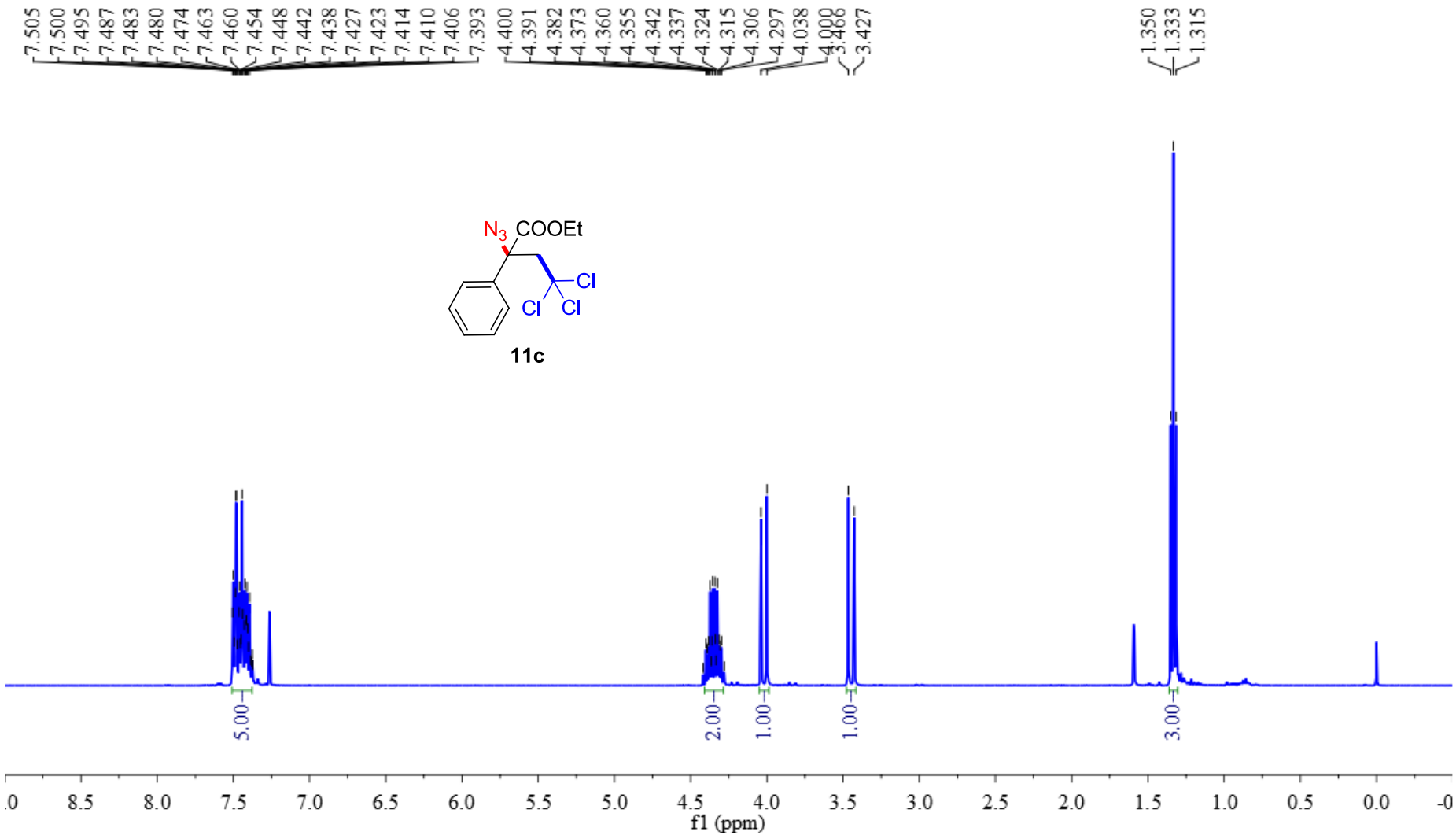
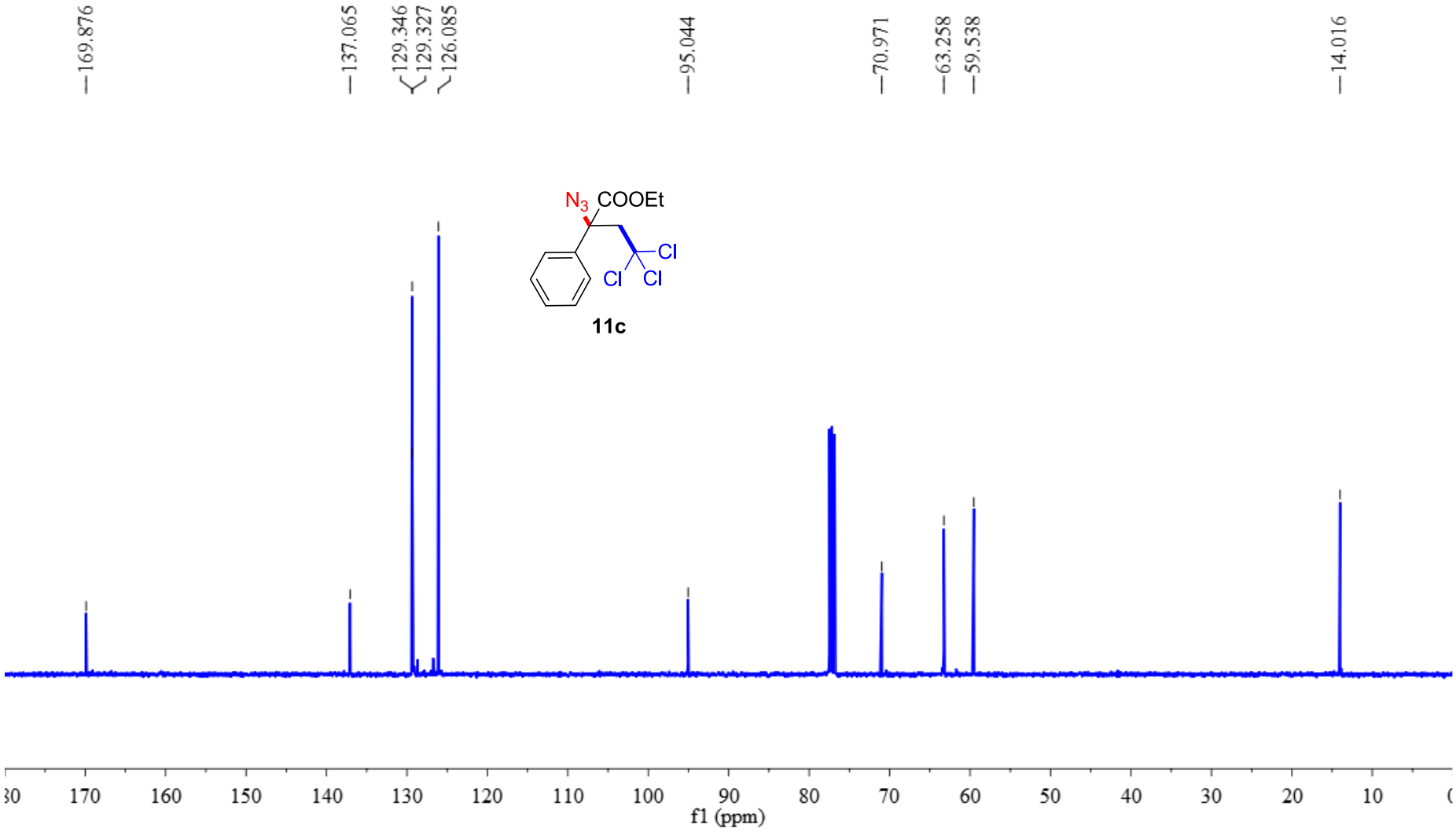
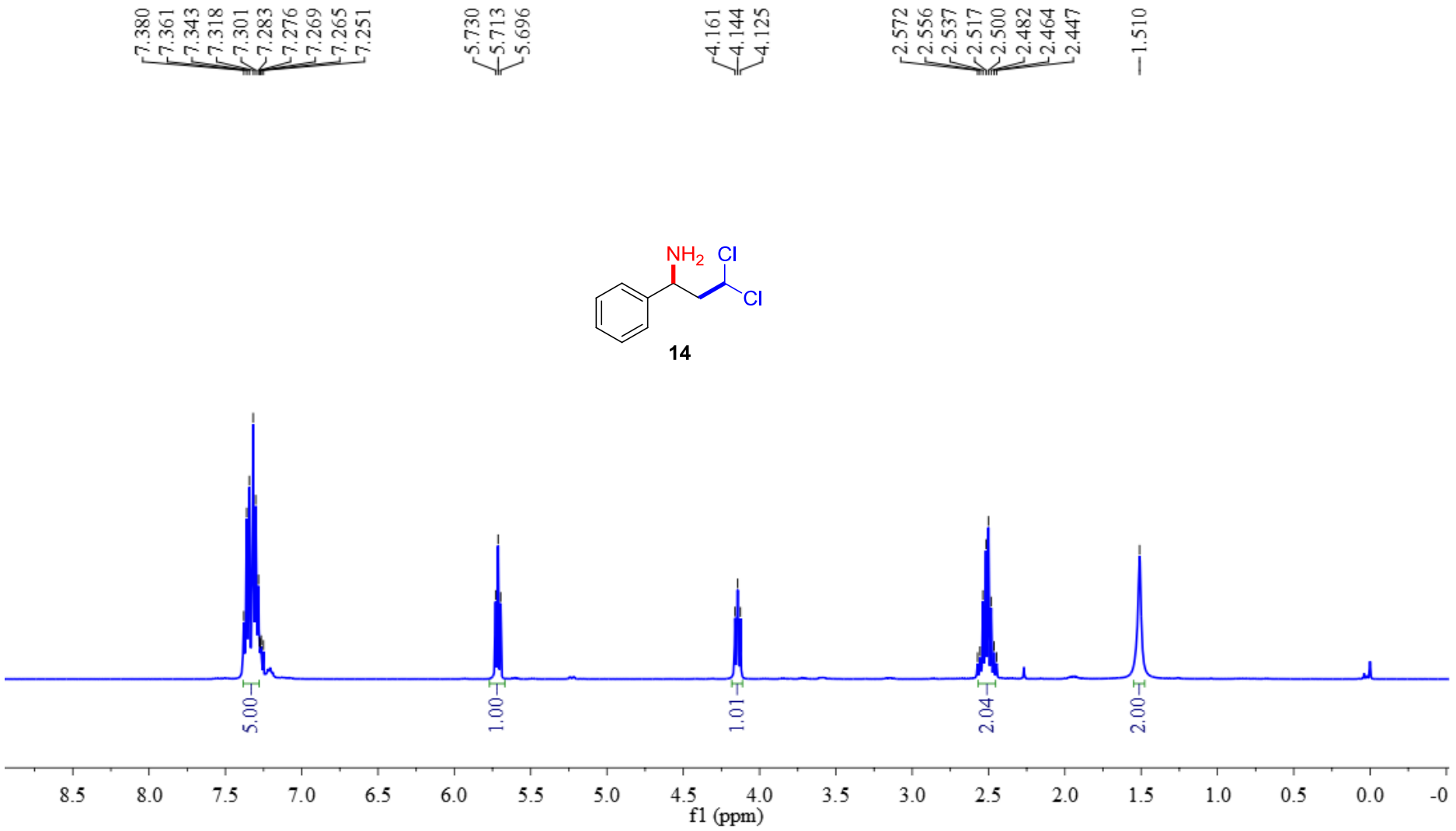
45.016
34.660
34.300
33.771
26.370
26.252
14.137
N3 COOEt
11a
f1 (ppm)

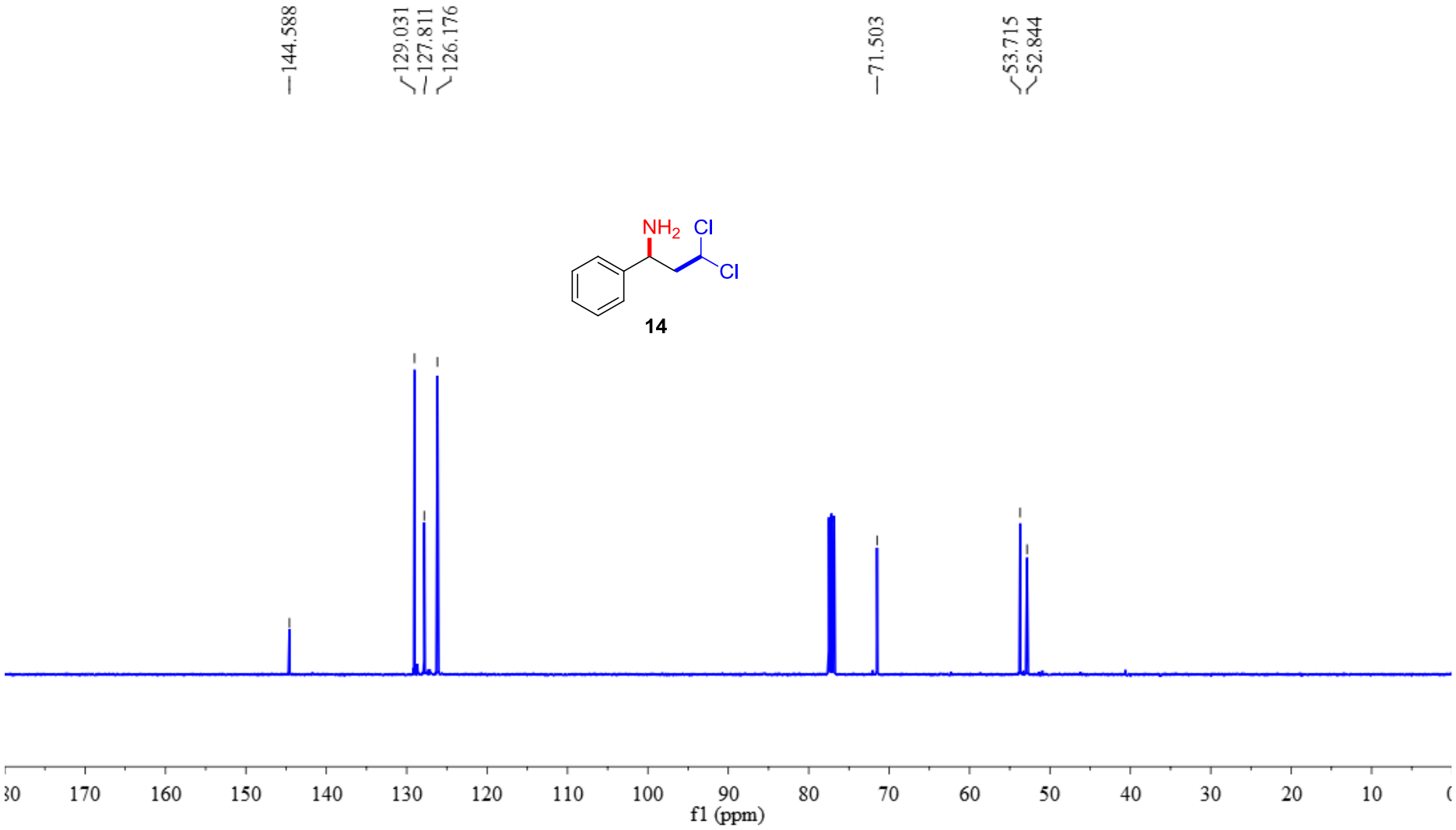

7.460
7.455
7.439
7.432
7.426
7.411
7.407
7.402
7.399
7.394
7.389
7.381
7.376
7.369
5.924
5.915
5.902
5.893
4.388
4.370
4.361
4.351
4.343
4.332
4.325
4.315
4.306
4.297
4.288
4.284
3.242
3.227
3.204
2.955
2.946
2.917
2.908
1.357
1.339
1.321
N3 COOEt
Cl
Cl
11b
5.01
1.00
2.01
1.01
1.01
3.00
f1 (ppm)

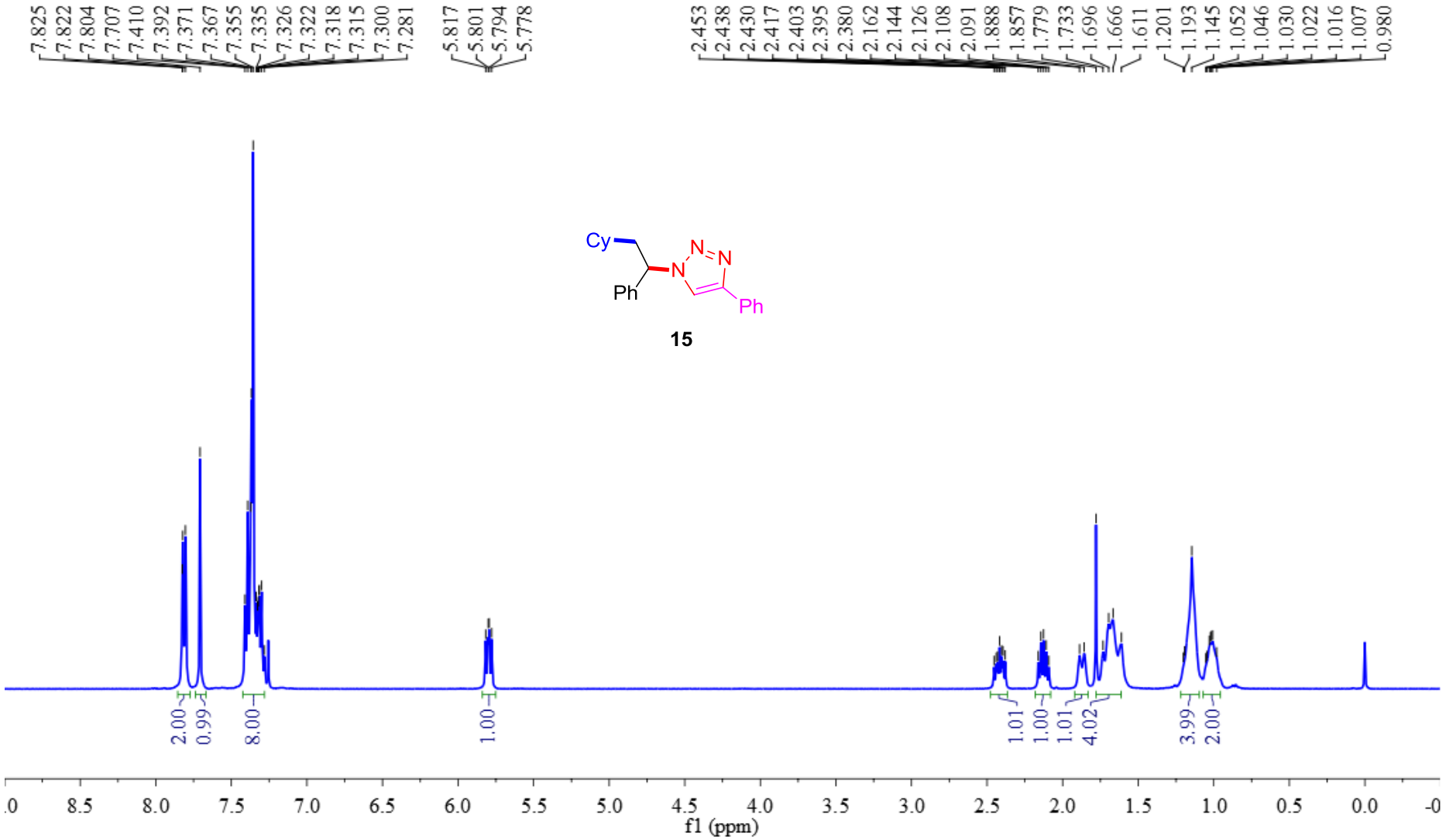

170.095
136.997
129.347
129.244
125.863
70.919
68.789
63.017
51.021
14.054
N3 COOEt
Cl
Cl
11b
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


7.505
7.500
7.495
7.487
7.483
7.480
7.474
7.463
7.460
7.454
7.448
7.442
7.438
7.427
7.423
7.414
7.410
7.406
7.393
4.400
4.391
4.382
4.373
4.360
4.355
4.342
4.337
4.324
4.315
4.306
4.297
4.038
4.000
3.468
3.427
1.350
1.333
1.315
N3 COOEt
Cl
Cl
Cl
11c
5.00
2.00
1.00
1.00
3.00
f1 (ppm)


169.876
137.065
129.346
129.327
126.085
95.044
70.971
63.258
59.538
14.016
N3
COOEt
Cl
Cl
Cl
11c
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


7.380
7.361
7.343
7.318
7.301
7.283
7.276
7.269
7.265
7.251
5.730
5.713
5.696
4.161
4.144
4.125
2.572
2.556
2.537
2.517
2.500
2.482
2.464
2.447
1.510
NH2
Cl
Cl
14
5.00
1.00
1.01
2.04
2.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)


144.588
129.031
127.811
126.176
71.503
53.715
52.844
NH2
Cl
Cl
14
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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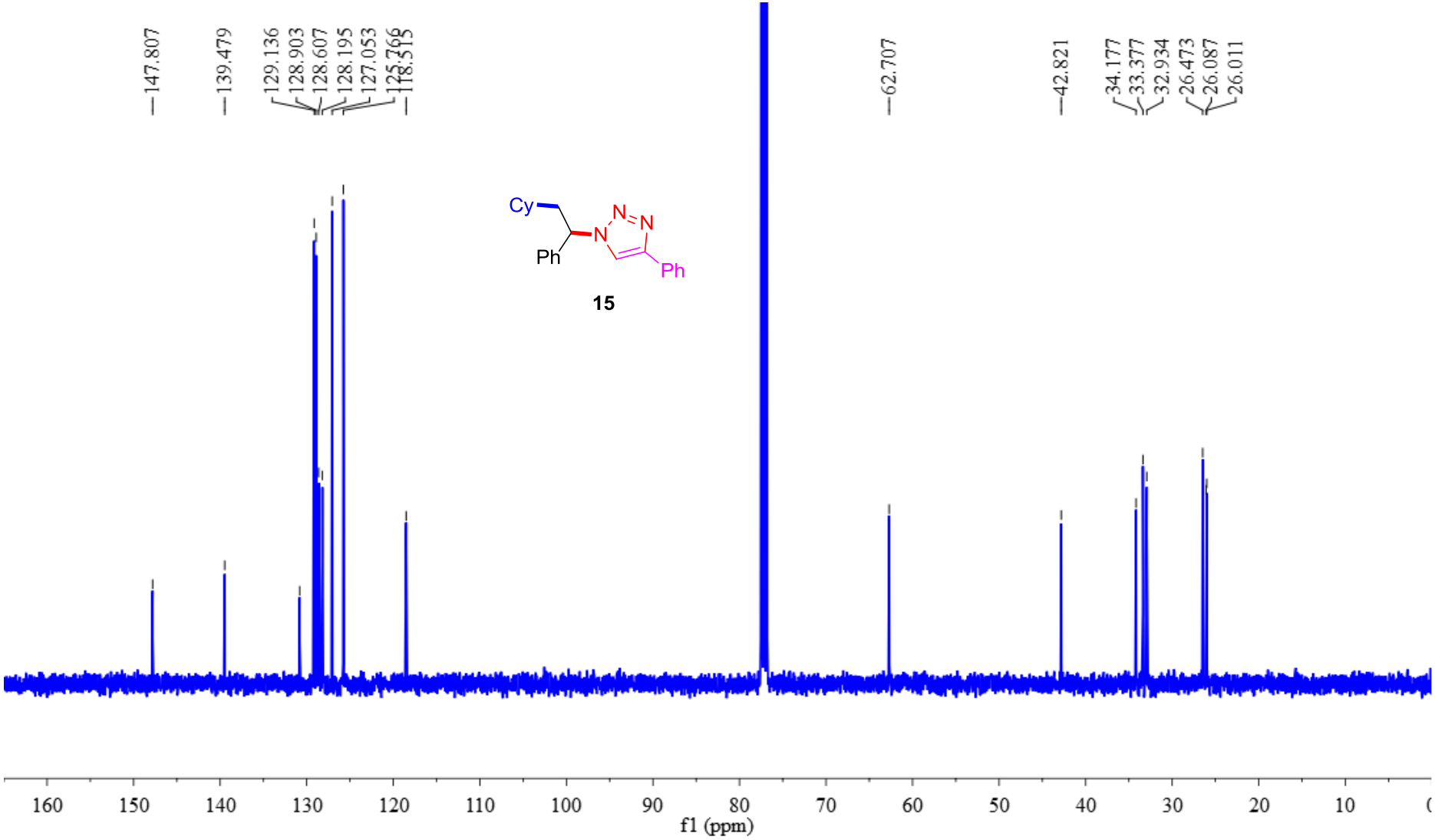
7.822
7.804
7.707
7.410
7.392
7.371
7.367
7.355
7.335
7.326
7.322
7.318
7.315
7.300
7.281
5.817
5.801
5.794
5.778
2.453
2.438
2.430
2.417
2.403
2.395
2.380
2.162
2.144
2.126
2.108
2.091
1.888
1.857
1.779
1.733
1.696
1.666
1.611
1.201
1.193
1.145
1.052
1.046
1.030
1.022
1.016
1.007
0.980
Cy
Ph
N
N
N
Ph
2.00
0.99
8.00
1.00
1.01
1.00
1.01
4.02
3.99
2.00
f1 (ppm)

15


147.807
139.479
129.136
128.903
128.607
128.195
127.053
125.766
118.515
62.707
42.821
34.177
33.377
32.934
26.473
26.087
26.011
Cy
Ph
N
N
N
Ph
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

15