

Electrode-switchable: exploring this new strategy to achieve regiodivergent azidoiodination of alkenes

Xin-Lei Sun,^a Chen-Xi Xia,^a Yue Ren,^{*a} Yu-Jin Li,^a Zhi-Qian Cao,^a Ling-Guo Meng^{*[a]}

[a] Key Laboratory of Green and Precise Synthetic Chemistry and Applications, Ministry of Education; School of Chemistry and Materials Science; Information College, Huaibei Normal University, Huaibei, Anhui 235000, P. R. China

* Corresponding author E-mail: renyue@chnu.edu.cn; menglg59@chnu.edu.cn

Table of Contents

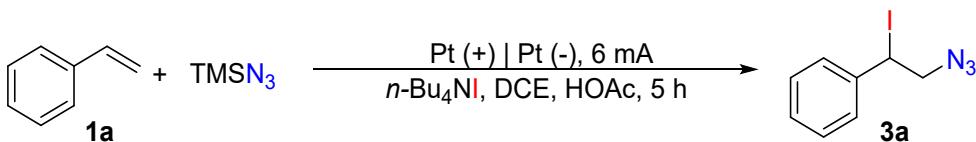
1. General remarks	S2
2. Optimization of Reaction Conditions.....	S2
3. General steps for synthesizing martensitic addition products from olefins.....	S4
4. General steps for synthesizing anti martensitic addition products from olefins.....	S5
5. Density functional theory.....	S6
6. CV experiments.....	S7
7. Characterization data for all products.....	S9
8. ¹ H NMR and ¹³ C NMR spectra of the products.....	S26
9. References.....	S75

1. General remarks

All reactions were conducted in clean glassware with magnetic stirring. Chromatographic purification was performed on silica gel (400~500 mesh) and analytical thin layer chromatography (TLC) on silica gel 60-F₂₅₄ (Qindao), which was detected by fluorescence. ¹H NMR (600 MHz) and ¹³C NMR (150 MHz) spectra were measured with a Bruker AC 600 spectrometer with CDCl₃ as solvent and recorded in ppm relative to internal tetramethylsilane standard. ¹H NMR data are reported as follows: δ, chemical shift; coupling constants (J are given in Hertz, Hz) and integration. Abbreviations to denote the multiplicity of a particular signal were s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), and br (broad singlet). High resolution mass spectra were obtained with a Thermo Scientific LTQ Orbitrap XL mass spectrometer (ESI). It should be noted that the molecular weight of some products is indirectly determined by further transformation into its Click products. Melting points were determined on a digital melting point apparatus and temperatures were uncorrected. HY3005MT potentiostat made by Huayi Company in China was used as a power supply device. All undivided cells were custom made by the Lianhua Glass Instrument Company in China. Carbon electrode is purchased from Lianhua Glass Instrument Company in China. Cyclic voltammetry (CV) measurement was conducted by using an electrochemical workstation (CHI 660E, Chenhua, China). Platinum electrode was purchased from China's Shang hai San She Instrument Company.

2. Optimization of Reaction Conditions

Table S1. Optimization conditions.^{a,b}



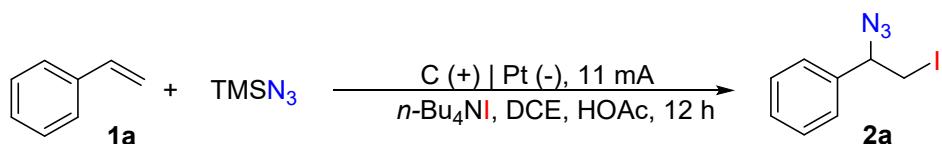
Entry	variation from standard conditions	3a yield (%)	2a yield (%)
1	None	66	/

2	0 mA	0	/
3	2 mA	N.R ^d	/
4	4 mA	36	/
5	8 mA	62	/
6	10 mA	48	/
7	3 h	50	/
8	7 h	58	/
9	<i>n</i> -Bu ₄ NI (0.4 mmol) instead of <i>n</i> -Bu ₄ NI (0.6 mmol)	48	/
10	<i>n</i> -Bu ₄ NI (0.8 mmol) instead of <i>n</i> -Bu ₄ NI (0.6 mmol)	34	/
11	TMSN ₃ (0.4 mmol) instead of TMSN ₃ (0.6 mmol)	58	/
12	TMSN ₃ (0.8 mmol) instead of TMSN ₃ (0.6 mmol)	32	/
13	Ni (+) Pt (-) instead of Pt (+) Pt (-)	12 ^c	/
14	Ni (+) Pt (-) instead of Pt (+) Pt (-)	20	/
15	C (+) C (-) instead of Pt (+) Pt (-)	N.R ^d	/
16	C (+) Ni (-) instead of Pt (+) Pt (-)	26	5
17	Pt (+) C (-) instead of Pt (+) Pt (-)	20	/
18	DCM instead of DCE	trace	/
19	CH ₃ OH instead of DCE	N.R ^d	/
20	CH ₃ CN instead of DCE	N.R ^d	/
21	DMSO instead of DCE	N.R ^d	/
22	DMF instead of DCE	N.R ^d	/
23	HOAc (0.3 mL)	34	/
24	HOAc (0.4 mL)	65	/
25	HOAc (0.6 mL)	58	/
26	Et ₄ NI instead of <i>n</i> -Bu ₄ NI	48	/
27	Me ₄ NI instead of <i>n</i> -Bu ₄ NI	N.R ^d	/
28	NaN ₃ instead of TMSN ₃	40	/
29	CH ₃ OH instead of HOAc	trace	/
30	HFIP instead of HOAc	58	/

^a Reaction conditions: **1a** (0.2 mmol), TMSN₃ (0.6 mmol), *n*-Bu₄NI (0.6 mmol, 3 equiv.), HOAc (0.5 mL) and DCE (6.0 mL) in an undivided cell with Pt plate as the anode and Pt plate as the

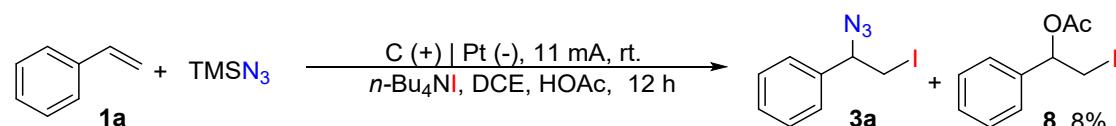
cathode, constant current =6 mA, 5 h. ^b Yield of the isolated product. ^c **1a** (0.2 mmol), TMSN₃ (0.6 mmol), Bu₄NI (0.6 mmol), HOAc (0.5 mL) and DCE (6.0 mL) in an undivided cell with a Ni plate as the anode and a Pt plate as the cathode, constant current = 11 mA, 12 h. ^dNR = No reaction.

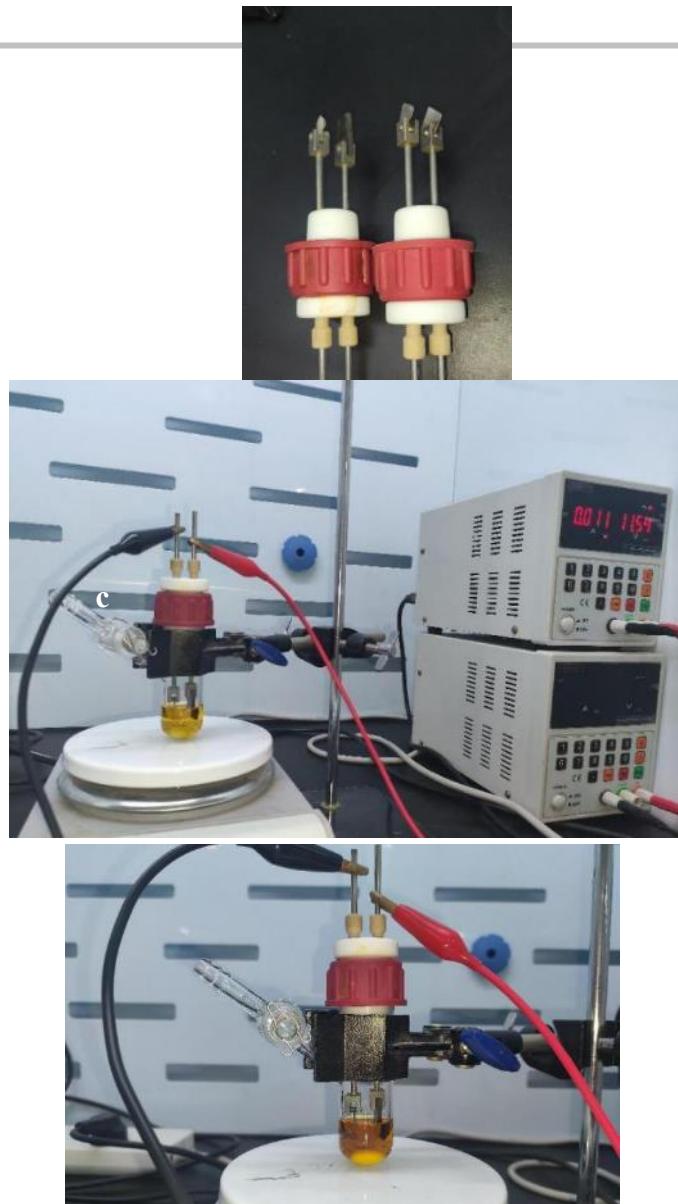
3. General steps for synthesizing Markovnikov's addition products from olefins



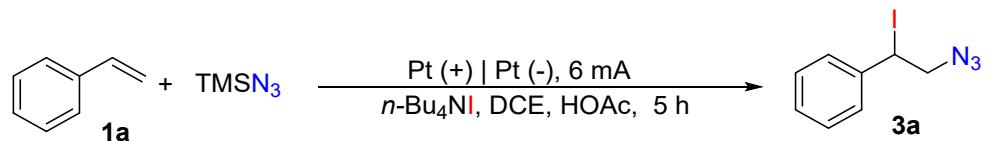
In an undivided electrolytic cell, the carbon plate (2.0 mm × 10.0 mm × 20.0 mm) is used as the anode and the platinum plate (0.1 mm × 10.0 mm × 10.0 mm) as the cathode, and connected to the direct current regulating power supply. Add olefins (0.2 mmol), TMSN₃ (0.6 mmol), HOAc (0.5 mL) and *n*-Bu₄NI (0.6 mmol) to the reaction vessel and dissolve them in DCE (6.0 mL). The mixture was subjected to constant current conditions (11 mA/cm²) under room temperature magnetic stirring, followed by reaction analysis using thin layer chromatography, and the current was removed. The mixture is subjected to vacuum evaporation to remove the solvent. The residue is purified by silica gel Column chromatography (Petroleum ether) to obtain the pure product.

Note: Under optimal reaction conditions, (1-acetate-2-iodoethyl)benzene with an 8% yield, aligning with structures documented in prior literature.¹



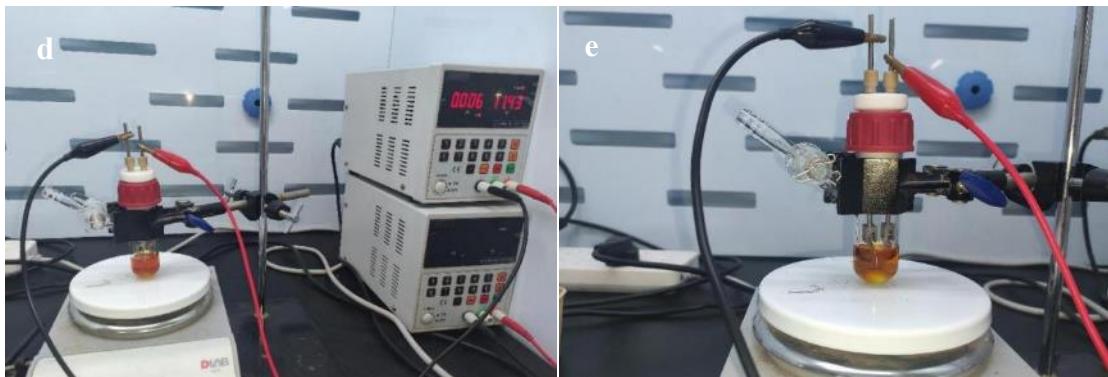


4. General steps for synthesizing anti-Markovnikov's addition addition products from olefins



In an undivided electrolytic cell, the platinum plate (0.1 mm × 10.0 mm × 10.0 mm) is used as the anode and the platinum plate (0.1 mm × 10.0 mm × 10.0 mm) as the cathode, and connected to the direct current regulating power supply. Add olefins (0.2 mmol), TMN₃ (0.6 mmol), HOAc (0.5 mL) and n-Bu₄NI (0.6 mmol) to the reaction vessel and dissolve them in DCE (6.0 mL). The

mixture was subjected to constant current conditions (12 mA/cm^2) under room temperature magnetic stirring, followed by reaction analysis using thin layer chromatography, and the current was removed. The mixture is subjected to vacuum evaporation to remove the solvent. The residue is purified by silica gel Column chromatography (Petroleum ether) to obtain the pure product.



5. Density functional theory:

To further compare the adsorption of I/N on the surface of carbon and Pt, the adsorption was calculated via DFT calculations with VASP. The projector augmented wave (PAW) method was used here to describe the interaction between the ion and electron, GGA-PBE was taken as the exchange-correlation functional and the plane-wave cut-off energy was set as 500 eV. Considering the computing resource and accuracy, the Gamma centered $5\times 5\times 1$ k-points mesh was built by Monkhorst–Pack method during structure optimization and the self-consistent calculation was performed with $13\times 13\times 13$ k-points mesh. The structures are optimized until the energy is converged to 1.0×10^{-5} eV/atom and the force converged to $0.01 \text{ eV}/\text{\AA}$.

The initial adsorption models of I/N on C and Pt are shown in Fig. S1. C model here consists of 50 atoms, while Pt (001) surface with two-layer atoms are selected as the adsorption model, where the bottom layer is fixed and the top layer can be relaxed. For the adsorption, the initial adsorption site is set as on the top of the chosen C or Pt atom and the adsorption distance is set as 3 Å. The adsorption models after optimization are shown in Fig. S2 and S3. For the adsorption of I on

C (Fig. S2), the optimized distance for C atom and I atom is 3.77 Å, which is larger than the initial distance. For the adsorption of I on Pt, the adsorption site transferred from the top of Pt atom to the middle of the two Pt atoms and the adsorption distance for I on Pt is 2.26 Å. Moreover, the adsorption energy for I on C and Pt is -0.56 eV and -2.81 eV, respectively, revealing that Pt possesses more excellent adsorption capability than that of C. The optimized N-adsorption models are shown in Fig. S3. For the adsorption of N, the C model possesses negligible adsorption capability, the adsorption distance and energy are 3.49 Å and -0.013 eV, respectively. In the Pt model, the adsorption distance and energy are 1.96 Å and -0.43 eV, respectively. Therefore, both for N and I, Pt exhibits superior adsorption capacity. Compared with N, Pt exhibit superior adsorption capacity for I.

6. CV experiments:

Figure S1. Cyclic voltammograms at carbon disc (2 mm in diameter) as work electrode, Pt plate ($1 \times 1 \text{ cm}^2$) as counter electrode and Ag/AgCl as reference electrode in 0.1 M $n\text{-Bu}_4\text{NBF}_4$, DCE (6 mL), scan rate: 0.1 V/s. (a) back-ground, 0.1 M $n\text{-Bu}_4\text{NBF}_4$; (b) $n\text{-Bu}_4\text{NI}$ (0.05 mmol); (c) TMSN_3 (0.05 mmol); (d) Styrene (0.05 mmol).

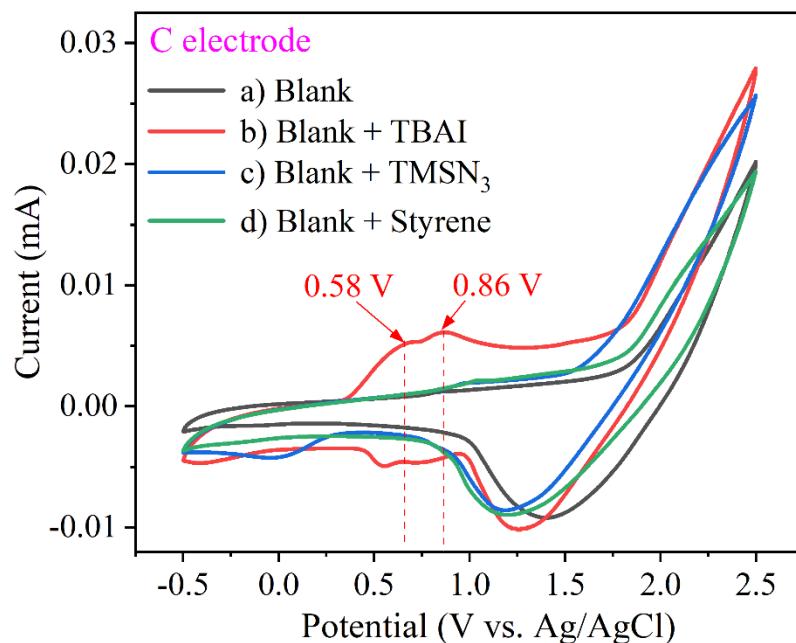


Figure S2. Cyclic voltammograms at platinum disc (2 mm in diameter) as work electrode, Pt plate ($1 \times 1 \text{ cm}^2$) as counter electrode and Ag/AgCl as reference electrode in 0.1 M $n\text{-Bu}_4\text{NBF}_4$, DCE (6 mL), scan rate: 0.1 V/s. (a) back-ground, 0.1 M $n\text{-Bu}_4\text{NBF}_4$; (b) $n\text{-Bu}_4\text{NI}$ (0.05 mmol); (c) TMSN_3 (0.05 mmol); (d) Styrene (0.05 mmol).

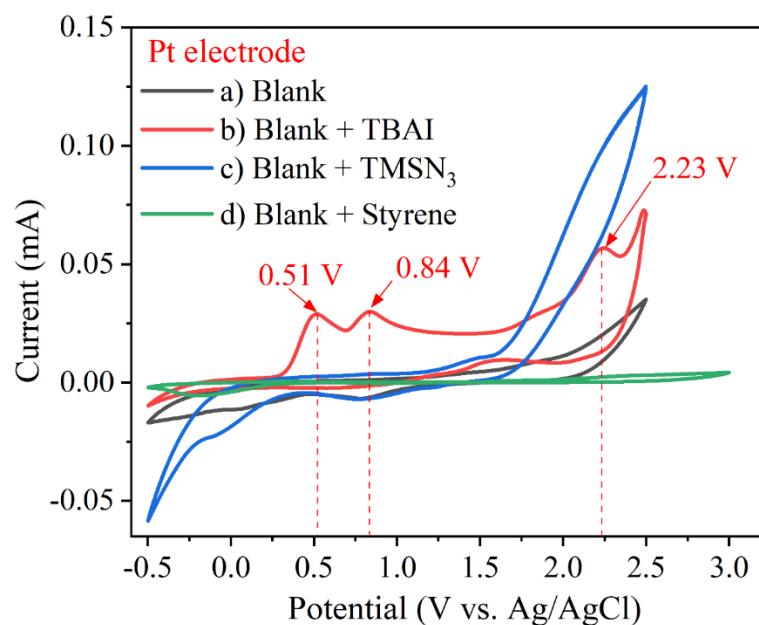
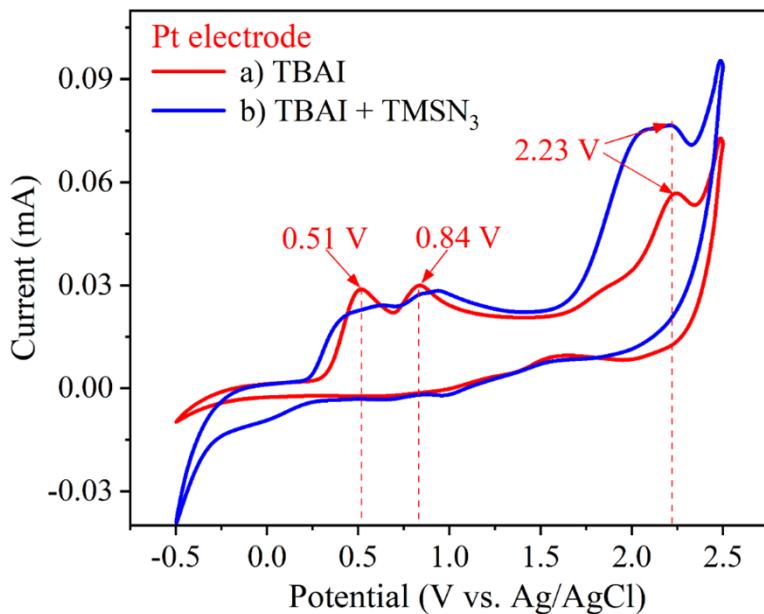


Figure S3. Cyclic voltammograms at platinum disc (2 mm in diameter) as work electrode, Pt plate ($1 \times 1 \text{ cm}^2$) as counter electrode and Ag/AgCl as reference electrode in 0.1 M $n\text{-Bu}_4\text{NBF}_4$, DCE (6 mL), scan rate: 0.1 V/s. (a) $n\text{-Bu}_4\text{NI}$ (0.05 mmol); (b) $n\text{-Bu}_4\text{NI}$ (0.05 mmol) + TMSN_3 (0.05 mmol).



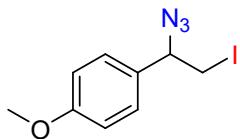
7. Characterization Data for All Products

(1-azido-2-iodoethyl) benzene (2a)²



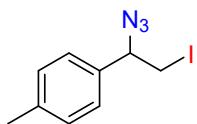
Colorless oil (36.0 mg, 66% yield). ¹H NMR (600 MHz, CDCl₃) δ 7.43-7.37 (m, 3H), 7.32 (d, *J* = 6.8 Hz, 2H), 4.72 (t, *J* = 7.0 Hz, 1H), 3.40 (d, *J* = 7.0 Hz, 2H); ¹³C NMR (150 MHz, CDCl₃) δ 137.9, 129.0, 129.0 126.6, 67.1, 8.1.

1-(1-azido-2-iodoethyl)-4-methoxybenzene (2b)²



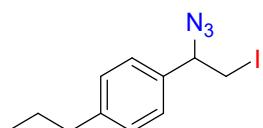
Pale yellow oil (39.9 mg, 66% yield). ¹H NMR (600 MHz, CDCl₃) δ 7.26-7.23(m, 2H), 6.94-6.92 (m, 2H), 4.68 (dd, *J* = 8.0, 6.2 Hz, 1H), 3.82 (s, 3H), 3.40-3.34 (m, 2H); ¹³C NMR (150 MHz, CDCl₃) δ 160.0, 129.8, 127.9, 114.3, 66.7, 55.3, 8.4.

1-(1-azido-2-iodoethyl)-4-methylbenzene (2c)²



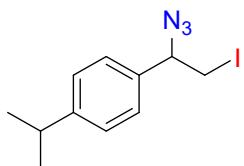
Colorless oil (35.5 mg, 62% yield). **$^1\text{H NMR}$** (600 MHz, CDCl_3) δ 7.24-7.20 (m, 4H), 4.69 (t, $J = 7.0$ Hz, 1H), 3.39 (d, $J = 7.1$ Hz, 2H), 2.38 (s, 3H); **$^{13}\text{C NMR}$** (150 MHz, CDCl_3) δ 138.9, 134.8, 129.7, 126.5, 66.9, 21.2, 8.2.

1-(1-azido-2-iodoethyl)-4-propylbenzene (2d)



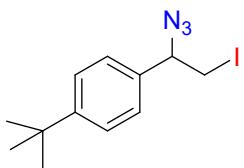
Colorless oil (29.0 mg, 46% yield). **$^1\text{H NMR}$** (600 MHz, CDCl_3) δ 7.23-7.20 (m, 2H), 4.69 (t, $J = 7.1$ Hz, 1H), 3.39 (d, $J = 7.1$ Hz, 2H), 2.60 (t, $J = 7.6$ Hz, 2H), 1.65 (td, $J = 14.9$ Hz, 7.4 Hz, 2H), 0.95 (t, $J = 7.3$ Hz, 3H); **$^{13}\text{C NMR}$** (150 MHz, CDCl_3) δ 143.8, 135.1, 129.1, 126.5, 67.1, 37.7, 24.3, 13.8, 8.3.

1-(1-azido-2-iodoethyl)-4-isopropylbenzene (2e)



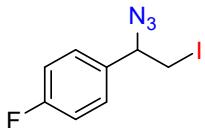
Colorless oil (29.3 mg, 45% yield). **$^1\text{H NMR}$** (600 MHz, CDCl_3) δ 7.27-7.22 (m, 2H), 4.69 (dd, $J = 7.8$ Hz, 6.2 Hz, 1H), 3.38-3.37 (m, 2H), 2.94-2.90 (m, 1H), 1.26 (dd, $J = 7.0$ Hz, 6H); **$^{13}\text{C NMR}$** (150 MHz, CDCl_3) δ 149.8, 135.3, 127.1, 126.6, 67.1, 33.8, 23.8, 8.3.

1-(1-azido-2-iodoethyl)-4-(tert-butyl) benzene (2f)²



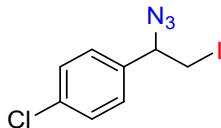
Pale yellow oil (39.5 mg, 60% yield). **¹H NMR** (600 MHz, CDCl₃) δ 7.43-7.41 (m, 2H), 7.26-7.24 (m, 2H), 4.70 (dd, J = 8.1, 6.0 Hz, 1H), 3.41- 3.36 (m, 2H), 1.32 (s, 9H); **¹³C NMR** (150 MHz, CDCl₃) δ 152.1, 134.9, 126.3, 125.9, 67.0, 34.7, 31.2, 8.3;

1-(1-azido-2-iodoethyl)-4-fluorobenzene (2g)²



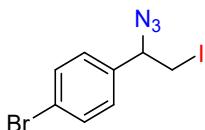
Colorless oil (23.3 mg, 40% yield). **¹H NMR** (600 MHz, CDCl₃) δ 7.30 (dd, J = 8.6, 5.2 Hz, 2H), 7.10 (t, J = 8.5 Hz, 2H), 4.71 (t, J = 6.2 Hz, 1H), 3.29-3.36 (m, 2H); **¹³C NMR** (150 MHz, CDCl₃) δ 162.8 (d, ¹J_{CF} = 246.9 Hz), 133.7 (d, ⁴J_{CF} = 3.2 Hz), 128.5 (d, ³J_{CF} = 8.5 Hz), 116.0 (d, ²J_{CF} = 21.7 Hz), 66.2, 8.0.

1-(1-azido-2-iodoethyl)-4-chlorobenzene (2h)²



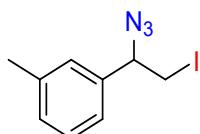
Colorless oil (23.3mg, 38% yield). **¹H NMR** (600 MHz, CDCl₃) δ 7.39 (d, J = 8.5 Hz, 2H), 7.26 (d, J = 8.4 Hz, 2H), 4.70 (t, J = 6.6 Hz, 1H), 3.37-3.35 (m, 2H); **¹³C NMR** (150 MHz, CDCl₃) δ 136.4, 134.9, 129.2, 128.0, 66.2, 7.7.

1-(1-Azido-2-iodoethyl)-4-bromobenzene (2i)³



Colorless oil (25.3 mg, 36% yield). **¹H NMR** (600 MHz, CDCl₃): δ 7.54 (d, J = 8.5 Hz, 2H) 7.20 (d, J = 8.4 Hz, 2H), 4.68 (t, J = 6.8 Hz, 1H), 3.37-3.35 (m, 2H); **¹³C NMR** (150 MHz, CDCl₃): δ 136.9, 132.2, 128.3, 123.1, 66.3, 7.6.

1-(1-azido-2-iodoethyl)-3-methylbenzene (2j)



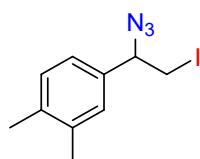
Colorless oil (35.0 mg, 61% yield). **¹H NMR** (600 MHz, CDCl₃) δ 7.30 (t, *J* = 7.4 Hz, 1H), 7.19 (d, *J* = 7.4 Hz, 1H), 7.12 (d, *J* = 7.7 Hz, 2H), 4.68 (t, *J* = 7.1 Hz, 1H), 3.39 (d, *J* = 7.3 Hz, 2H), 2.39 (s, 3H); **¹³C NMR** (150 MHz, CDCl₃) δ 138.8, 137.8, 129.8, 128.9, 127.3, 123.7, 67.3, 21.4, 8.2.

1-(1-Azido-2-iodoethyl)-2-methylbenzene (2k)³



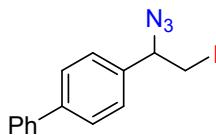
Colorless oil (33.2 mg, 58% yield). **¹H NMR** (600 MHz, CDCl₃) δ 7.34-7.33 (m, 1H), 7.27-7.24 (m, 2H), 7.21-7.20 (m, 1H), 4.95 (dd, *J* = 8.2, 5.9 Hz, 1H), 3.40-3.35 (m, 2H), 2.40 (s, 3H); **¹³C NMR** (150 MHz, CDCl₃) δ 136.1, 135.3, 131.0, 128.7, 126.7, 125.7, 63.7, 19.3, 7.0.

4-(1-azido-2-iodoethyl)-1,2-dimethylbenzene (2l)



Colorless oil (27.1 mg, 45% yield). **¹H NMR** (600 MHz, CDCl₃) δ 7.16(d, *J* = 7.7 Hz, 1H), 7.07-7.03(m, 2H), 4.65(t, *J* = 7.1 Hz, 1H), 3.37(d, 7.1 Hz, 2H), 2.28(d, *J* = 10.9 Hz, 6H); **¹³C NMR** (150 MHz, CDCl₃) δ 137.7, 137.4, 135.2, 130.2, 127.8, 124.0, 67.1, 19.9, 19.6, 8.3.

4-(1-azido-2-iodoethyl)-1,1'-biphenyl (2m)²



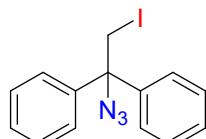
Yellow oil (36.3 mg, 53% yield). **¹H NMR** (600 MHz, CDCl₃) δ 7.65-7.59 (m, 4H), 7.47-7.37 (m, 5H), 4.77 (t, J = 7.1 Hz, 1H), 3.44-3.43 (m, 2H); **¹³C NMR** (150 MHz, CDCl₃) δ 142.0, 140.2, 136.8, 128.9, 127.7, 127.7, 127.1, 66.9, 8.0.

2-(1-azido-2-iodoethyl) naphthalene (2n)²



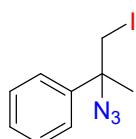
Yellow oil (30.4 mg, 47% yield). **¹H NMR** (600 MHz, CDCl₃) δ 7.91-7.86 (m, 3H), 7.80 (s, 1H), 7.55-7.53 (dd, J = 5.6, 4.92 Hz, 2H), 7.42 (dd, J = 8.5, 1.8 Hz, 1H), 4.90 (t, J = 6.6 Hz, 1H), 3.48 (d, J = 6.0 Hz, 2H); **¹³C NMR** (150 MHz, CDCl₃) δ 135.1, 133.4, 133.0, 129.1, 128.1, 127.8, 126.7, 126.3, 123.6, 67.3, 8.0.

(1-azido-2-iodoethane-1,1-diy) dibenzene (2o)²



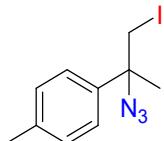
Colorless oil (41.2 mg, 59% yield). **¹H NMR** (600 MHz, CDCl₃) δ 7.44-7.33 (m, 10H), 4.10 (s, 2H); **¹³C NMR** (150 MHz, CDCl₃) δ 140.6, 128.5, 128.2, 127.2, 71.2, 15.6.

(2-azido-1-iodopropan-2-yl) benzene (2p)²



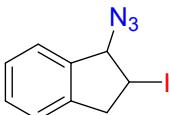
Colorless oil (38.5 mg, 67% yield). **¹H NMR** (600 MHz, CDCl₃) δ 7.46-7.7.33 (m, 5H), 3.49 (q, J = 10.6 Hz, 2H), 1.90 (s, 3H); **¹³C NMR** (150 MHz, CDCl₃) δ 140.7, 128.8, 128.2, 125.6, 65.0, 25.1, 17.7.

1-(2-azido-1-iodopropan-2-yl)-4-methylbenzene (2q)



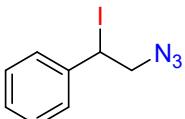
Colorless oil (38.5 mg, 64% yield). **¹H NMR** (600 MHz, CDCl₃) δ 7.36-7.31 (d, J = 8.3 Hz, 2H), 7.24-7.20 (d, J = 8.1 Hz, 2H), 3.5 (q, J = 10.62, 3H), 2.4 (s, 3H); 1.9 (s, 3H); **¹³C NMR** (150 MHz, CDCl₃) δ 138.1, 137.6, 129.4, 125.5, 65.0, 25.1, 20.1, 17.9.

1-azido-2-iodo-2,3-dihydro-1H-indene (2r)²



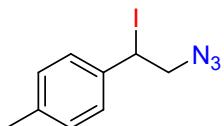
Colorless oil (18.2 mg, 32% yield). **¹H NMR** (600 MHz, CDCl₃) δ 7.42-7.26 (m, 4H), 5.12 (d, J = 5.5 Hz, 1H), 4.37 (q, J = 6.5 Hz, 1H), 3.64 (dd, J = 17.0, 7.0 Hz, 1H), 3.34 (dd, J = 16.6, 6.4 Hz, 1H); **¹³C NMR** (150 MHz, CDCl₃) δ 141.2, 138.5, 129.4, 127.6, 124.7, 124.4, 74.9, 43.5, 25.1.

(2-azido-1-iodoethyl) benzene (3a)²



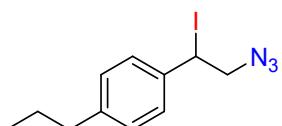
Colorless oil (36.0 mg, 66% yield). **¹H NMR** (600 MHz, CDCl₃) δ 7.43 (d, J = 7.3 Hz, 2H), 7.36-7.30 (m, 3H), 5.15 (t, J = 7.7 Hz, 1H), 3.94 (d, J = 7.7 Hz, 2H); **¹³C NMR** (150 MHz, CDCl₃) δ 140.3, 129.0, 128.8, 127.6, 58.8, 27.8.

1-(2-Azido-1-iodoethyl)-4-methylbenzene (3b)⁴



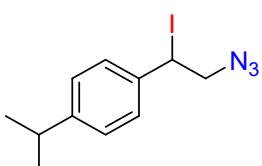
Colorless oil (41.3 mg, 72% yield). **¹H NMR** (600 MHz, CDCl₃): δ 7.32 (d, *J* = 8.2 Hz, 2H), 7.15 (d, *J* = 7.9 Hz, 2H), 5.15 (t, *J* = 7.8 Hz, 1H), 3.93 (d, *J* = 7.8 Hz, 2H), 2.33 (s, 3H); **¹³C NMR** (150 MHz, CDCl₃): δ 138.8, 137.3, 129.7, 127.4, 58.7, 28.2, 21.2.

1-(2-azido-1-iodoethyl)-4-propylbenzene(3c)



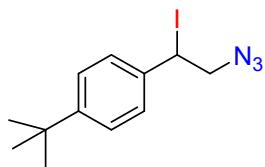
Colorless oil (31.5 mg, 50% yield). **¹H NMR** (600 MHz CDCl₃) δ 7.33 (d, *J* = 8.1 Hz, 2H), 7.15 (d, *J* = 8.0 Hz, 2H), 5.16 (t, *J* = 7.7 Hz, 1H), 3.93 (d, *J* = 7.7 Hz, 2H), 2.57 (t, *J* = 8.9 Hz, 2H), 1.67-1.61 (m, 2H), 0.94 (t, *J* = 7.3 Hz, 3H); **¹³C NMR** (150 MHz CDCl₃) δ 143.6, 137.5, 129.1, 127.4, 58.8, 37.7, 28.4, 24.2, 13.8.

1-(2-azido-1-iodoethyl)-4-isopropylbenzene (3d)



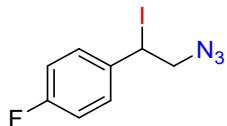
Colorless oil (33.4 mg, 53% yield). **¹H NMR** (600 MHz CDCl₃) δ 7.35 (d, *J* = 8.2 Hz, 2H), 7.19 (d, *J* = 8.2 Hz, 2H), 5.16 (t, *J* = 8.0 Hz, 1H), 3.94-3.92 (m, 2H), 2.92-2.87 (m, 1H), 1.25 (d, *J* = 6.9, 6H); **¹³C NMR** (150 MHz CDCl₃) δ 149.7, 137.5, 127.4, 127.1, 58.8, 33.8, 28.4, 23.7.

1-(2-azido-1-iodoethyl)-4-(tert-butyl) benzene (3e)⁵



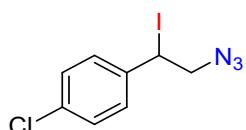
Pale yellow oil (40.1 mg, 61% yield). **¹H NMR** (600 MHz CDCl₃) δ 7.36 (s, 2H), 7.29 (d, *J* = 8.2 Hz, 2H), 5.18 (t, *J* = 8.1 Hz, 1H), 3.97-3.90 (m, 2H), 1.32 (s, 9H); **¹³C NMR** (150 MHz CDCl₃) δ 151.9, 137.1, 127.2, 125.9, 58.8, 34.7, 31.2, 28.3.

1-(2-azido-1-iodoethyl)-4-fluorobenzene (3f)⁵



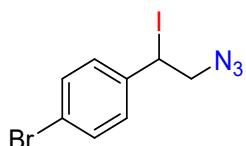
Colorless oil (22.1 mg, 38% yield). **¹H NMR** (600 MHz CDCl₃) δ 7.43-7.40 (m, 2H), 7.03 (t, *J* = 8.6 Hz, 2H), 5.13 (t, *J* = 8.6 Hz, 1H), 3.95-3.88 (m, 2H); **¹³C NMR** (150 MHz CDCl₃) δ 162.5 (d, ¹J_{CF} = 247.4 Hz), 136.2 (d, ⁴J_{CF} = 3.4 Hz), 129.4 (d, ³J_{CF} = 8.3 Hz), 116.0 (d, ²J_{CF} = 21.7 Hz), 58.9, 26.5.

1-(2-Azido-1-iodoethyl)-4-chlorobenzene (3g)⁴



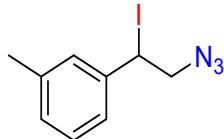
Colorless oil (23.3 mg, 38% yield). **¹H NMR** (600 MHz, CDCl₃): δ = 7.38-7.31 (m, 4H), 5.11 (dd, *J* = 8.8, 6.7 Hz, 2H), 3.95-3.88 (m, 2H); **¹³C NMR** (150 MHz, CDCl₃): δ 138.9, 134.4, 129.2, 128.9, 58.6, 26.2.

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



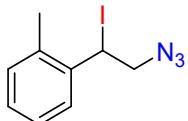
Pale yellow oil (31.6 mg, 45% yield). **¹H NMR** (600 MHz, CDCl₃): δ 7.47 (d, *J* = 8.3, 2H), 7.30 (d, *J* = 8.3, 2H), 5.09 (t, *J* = 8.6, 1H), 3.95-3.88 (m, 2H); **¹³C NMR** (150 MHz, CDCl₃): δ 139.4, 132.2, 129.2, 122.6, 58.5, 26.2.

1-(2-azido-1-iodoethyl)-3-methylbenzene (3i)



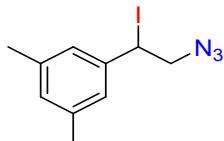
Pale yellow oil (33.3 mg, 58% yield). **¹H NMR** (600 MHz, CDCl₃): δ 7.23 (d, *J* = 3.24 Hz, 3H), 7.13-7.11 (m, 1H), 5.13 (t, *J* = 7.7 Hz, 1H), 3.93 (d, *J* = 7.7 Hz, 2H), 2.36 (s, 3H) **¹³C NMR** (150 MHz, CDCl₃): δ 140.1, 138.8, 129.6, 128.9, 128.2, 124.6, 58.7, 28.1, 21.3.

1-(2-azido-1-iodoethyl)-2-methylbenzene (3j)



Pale yellow oil (26.1 mg, 49% yield). **¹H NMR** (600 MHz, CDCl₃): δ 7.46 (d, *J* = 7.5 Hz, 1H), 7.24-7.18 (m, 2H), 7.13 (d, *J* = 7.3 Hz, 1H), 5.34 (t, *J* = 7.8 Hz, 1H), 4.05-3.99 (m, 2H), 2.34 (s, 3H); **¹³C NMR** (150 MHz, CDCl₃): δ 138.1, 135.3, 131.1, 128.6, 126.9, 126.7, 57.9, 24.6, 19.2.

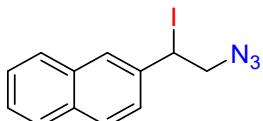
1-(2-azido-1-iodoethyl)-3,5-dimethylbenzene (3k)



Pale yellow oil (21.6 mg, 38% yield). **¹H NMR** (600 MHz, CDCl₃): δ 7.03 (s, 2H), 6.93 (s, 1H), 5.09 (t, *J* = 7.8 Hz, 1H), 3.92 (d, *J* = 7.6 Hz, 2H), 2.3 (s, 6H); **¹³C NMR** (150 MHz, CDCl₃): δ 140.0, 138.6, 130.6, 125.2, 58.7, 28.4, 21.2; **HRMS**

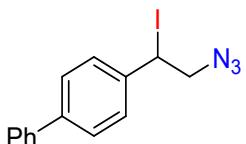
(ESI): calcd For C₁₀H₁₂IN⁺ (M+H-N₂)⁺: 273.0009, found 273.0006.

2-(2-azido-1-iodoethyl) naphthalene (3l)²



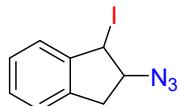
Yellow oil (24 mg, 37% yield). **¹H NMR** (600 MHz, CDCl₃) δ 7.86-7.82 (m, 4H), 7.55-7.50 (m, 3H), 5.36 (t, J = 7.7 Hz, 1H), 4.05 (d, J = 7.7 Hz, 2H); **¹³C NMR** (150MHz, CDCl₃) δ 137.4, 133.2, 133.0, 129.1, 128.0, 127.8, 126.8, 126.7, 126.3, 125.1, 58.5, 28.5.

4-(1-azido-2-iodoethyl)-1,1'-biphenyl (3m)³



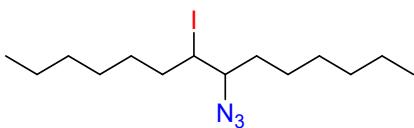
Pale yellow oil (27.9 mg, 40% yield). **¹H NMR** (600 MHz, CDCl₃) δ 7.61-7.57 (m, 4H), 7.51-7.36 (m, 5H), 5.22 (t, J = 7.9 Hz, 1H), 4.00-3.98 (m, 2H); **¹³C NMR** (150 MHz, CDCl₃) δ 141.7, 140.2, 139.2, 128.8, 128.0, 127.7, 127.6, 127.0, 58.7, 27.7.

2-azido-1-iodo-2,3-dihydro-1H-indene (3n)²



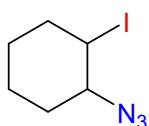
Colorless oil (35.3 mg, 62% yield); **¹H NMR** (600 MHz, CDCl₃) δ 7.42-7.41 (m, 1H), 7.27-7.23 (m, 3H), 5.19 (d, J = 1.38 Hz, 1H), 4.57 (dt, J = 5.7, 2.0 Hz, 1H), 3.40 (dd, J = 16.5, 5.8 Hz, 1H), 2.93 (dd, J = 16.4, 1.6 Hz, 1H); **¹³C NMR** (150 MHz, CDCl₃) δ 142.6, 139.5, 129.1, 128.0, 125.6, 125.1, 70.8, 36.5, 30.5.

7-azido-8-iodotetradecane (2s)



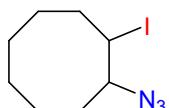
Colorless oil. **¹H NMR** (600 MHz, CDCl₃) δ 4.14-4.09 (m, 1H), 3.33-3.01(m, 1H), 1.76-1.50 (m, 6H), 1.34-1.28 (m, 14H), 0.90-0.88 (m, 6H); **¹³C NMR** (150 MHz, CDCl₃) δ 67.9, 66.5, 41.1, 40.7, 37.3, 35.7, 34.2, 33.5, 31.6, 31.5, 29.6, 29.6, 28.9, 28.9, 28.4, 28.4, 26.2, 26.0, 22.6, 22.5, 22.5, 14.0; **HRMS (ESI)**: C₁₄H₂₉IN⁺ (M+H-N₂)⁺ 338.1339, found 338.1340.

1-azido-2-iodocyclohexane (2t)⁴



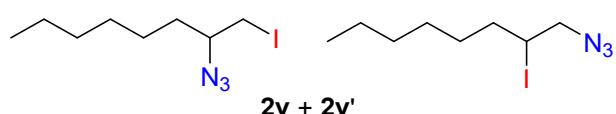
Colorless oil. **¹H NMR** (600 MHz, CDCl₃) δ 3.96 (td, J = 10.7, 4.1 Hz, 1H), 3.56-3.46 (m, 1H), 2.50-2.38 (m, 1H), 2.22-2.12 (m, 1H), 2.06-1.95 (m, 1H), 1.93-1.83 (m, 1H), 1.61-1.54 (m, 1H), 1.49-1.37 (m, 2H), 1.34-1.24 (m, 1H); **¹³C NMR** (150 MHz, CDCl₃) δ 67.2, 38.4, 33.2, 31.9, 27.0, 23.8.

1-Azido-2-iodocyclooctane (2u)⁶



Colorless oil. **¹H NMR** (600 MHz, CDCl₃) δ 4.66-4.57 (m, 1H), 4.30-4.22 (m, 1H), 3.90 (t, J = 8.2 Hz, 1H), 3.50-3.41 (m, 1H), 1.33-2.41 (m, 12H); **¹³C NMR** (150 MHz, CDCl₃) δ 71.2, 64.2, 39.3, 38.9, 37.0, 34.2, 31.7, 31.5, 27.2, 26.2, 26.2, 25.6, 25.2, 24.0.

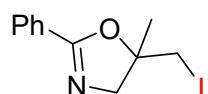
1-azido-2-iodooctane + 2-azido-1-iodooctane (2v+2v')⁷



2v: Colorless oil. **¹H NMR** (600 MHz, CDCl₃): δ = 3.42-3.38 (m, 1H), 3.29-3.23 (m, 2H), 1.71-1.66 (m, 1H), 1.60-1.54 (m, 1H), 1.39-1.29 (m, 8H), 0.89 (t, J = 6.8 Hz, 3H); **¹³C NMR** (150 MHz, CDCl₃): δ = 62.7, 34.4, 31.6, 28.8, 25.8, 22.5, 14.0, 8.5.

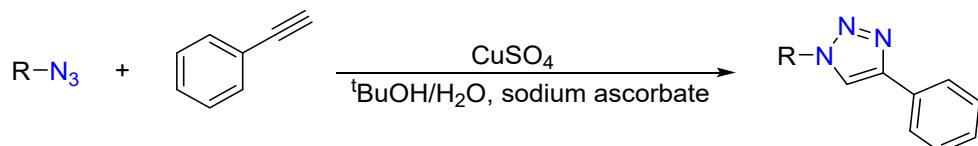
2v': Colorless oil. **¹H NMR** (600 MHz, CDCl₃): δ = 4.11-4.07 (m, 1H), 3.74 (dd, J = 12.9, 6.12 Hz, 1H), 3.63 (dd, J = 12.9, J = 6.9 Hz, 1H), 1.80-1.75 (m, 2H), 1.46-1.39 (m, 8 H), 0.88 (t, J = 6.8 Hz, 3H); **¹³C NMR** (150 MHz, CDCl₃): δ = 59.0, 37.1, 32.3, 31.5, 29.1, 28.4, 22.5, 14.0.

5-(iodomethyl)-5-methyl-2-phenyl-4,5-dihydrooxazole (6)⁸



Pale yellow oil (48.1 mg, 80% yield). **¹H NMR** (600 MHz, CDCl₃) δ 7.93(d, J = 7.1 Hz, 2H), 7.47(t, J = 7.4 Hz, 1H), 7.41(t, J = 7.8 Hz, 2H), 4.01(d, J = 15 Hz, 1H), 3.85(d, J = 14.9 Hz, 1H), 3.42(dd, J = 24.0, 10.5 Hz, 2H), 1.67(s, 3H); **¹³C NMR** (150 MHz, CDCl₃) δ 162.9, 131.4, 128.3, 128.1, 127.7, 83.7, 65.5, 25.3, 14.0.

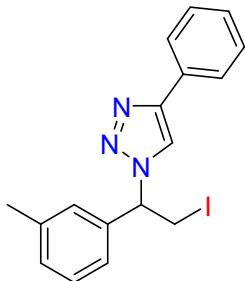
6.1 Synthesis steps of triazole compounds⁹



Compound **2/3**, (0.2 mmol, 1 equiv.), CuSO₄ (10 mg, 0.619 mmol, 0.2 equiv.), Phenylacetylene (0.24 mmol, 1.2 equiv.), sodium ascorbate (24 mg, 0.123 mmol, 0.4 equiv.) were suspended in ^tBuOH: H₂O (2 mL:1mL) (2:1) and the reaction mixture was stirred at room temperature for 3h. The reaction mixture was diluted with EtOAc (10 mL), quenched with saturated NaHCO₃ (5 mL) and extracted with EtOAc (3 × 30 mL). The combined organic layers were washed with brine solution, dried over anhydrous Na₂SO₄, concentrated in

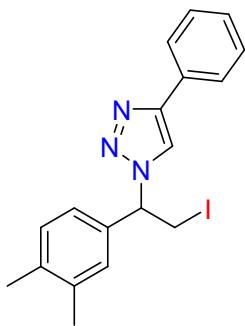
vacuo and purified by silica gel column chromatography (Hexane/EtOAc: from 10:1 to 10:2) to afford the triazole **7** as white solid.

1-(2-iodo-1-(m-tolyl) ethyl)-4-phenyl-1H-1,2,3-triazole (7a)



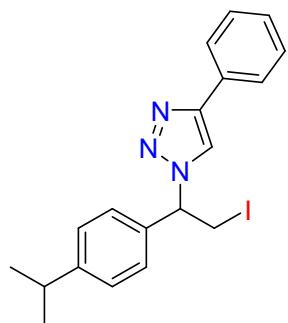
White solid. Mp: 163-165 °C. **1H NMR** (600 MHz, CDCl₃) δ 7.90 (d, *J* = 7.1 Hz, 2H), 7.80 (s, 1H), 7.47 (t, *J* = 7.5 Hz, 2H), 7.40-7.32 (m, 2H), 7.25 (t, *J* = 2.7 Hz, 3H), 5.8 (dd, *J* = 9.2, 6.1 Hz, 1H), 4.3 (dd, *J* = 10.7, 9.2 Hz, 1H), 3.92 (dd, *J* = 10.8, 6.1 Hz, 1H), 2.42 (s, 3H); **13C NMR** (150 MHz, CDCl₃) δ 147.6, 139.2, 137.1, 130.4, 130.1, 129.1, 128.8, 128.2, 127.5, 125.7, 123.9, 119.5, 67.1, 21.4, 5.0; **HRMS (ESI)**: calcd For C₁₇H₁₇IN₃⁺ (M+H)⁺: 390.0462; Found: 390.0464.

1-(1-(3,4-dimethylphenyl)-2-iodoethyl)-4-phenyl-1H-1,2,3-triazole (7b)



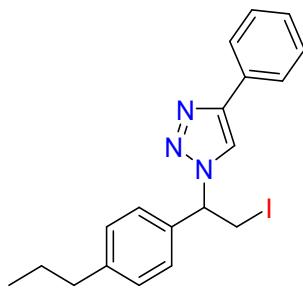
19.5, 5.2; **HRMS (ESI)**: calcd For $C_{18}H_{19}IN_3^+$ ($M+H$)⁺ : 404.0618; Found: 404.0619.

1-(2-iodo-1-(4-isopropylphenyl) ethyl)-4-phenyl-1H-1,2,3-triazole (7c)



White solid. Mp:135-137 °C. **1H NMR** (600 MHz, $CDCl_3$) δ 7.84-7.82 (m, 2H), 7.75 (s, 1H), 7.40 (t, J = 7.5 Hz, 2H), 7.33-7.30 (m, 3H), 7.24 (d, J = 8.2 Hz, 2H), 5.76 (dd, J = 9.4, 5.9 Hz, 1H), 4.23 (dd, J = 12.5, 9.4 Hz, 1H), 3.86 (dd, J = 10.8, 5.9 Hz, 1H), 2.93-2.88 (m, 1H), 1.24 (d, J = 6.9 Hz, 6H); **13C NMR** (150 MHz, $CDCl_3$) δ 150.2, 147.6, 134.5, 130.4, 128.8, 128.2, 127.3, 126.8, 125.7, 119.5, 66.9, 33.8, 23.8, 5.2; **HRMS (ESI)**: calcd For $C_{19}H_{21}IN_3^+$ ($M+H$)⁺ : 418.0775; Found: 418.0772.

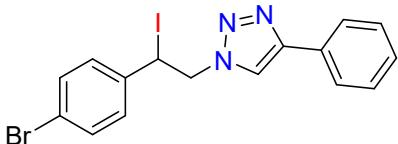
1-(2-iodo-1-(4-propylphenyl) ethyl)-4-phenyl-1H-1,2,3-triazole (7d)



White solid. Mp:129-131 °C. **1H NMR** (600 MHz, $CDCl_3$) δ 7.84-7.82 (m, 2H), 7.76 (s, 1H), 7.40 (t, J = 7.5 Hz, 2H), 7.33-7.29 (m, 3H), 7.20 (d, 8.2 Hz, 2H), 5.76 (dd, J = 7.2, 6.0 Hz, 1H), 4.22 (dd, J = 10.7, 9.3 Hz, 1H), 3.85 (dd, J = 10.7, 6.0 Hz, 1H), 2.58 (t, J = 7.5 Hz, 2H), 1.66-1.59 (m, 2H), 0.93 (t, J = 7.3 Hz,

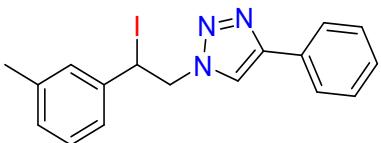
3H); **¹³C NMR** (150 MHz, CDCl₃) δ 146.6, 143.1, 133.4, 129.4, 128.3, 127.8, 127.2, 125.7, 124.7, 118.5, 65.9, 36.6, 23.3, 12.8, 4.3; **HRMS (ESI)**: calcd For C₁₉H₂₁IN₃⁺ (M+H)⁺: 418.0775; Found: 418.0775.

1-(2-(4-bromophenyl)-2-iodoethyl)-4-phenyl-1H-1,2,3-triazole (7e)



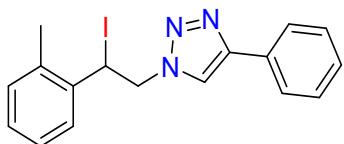
White solid. Mp: 155-157 °C. **¹H NMR** (600 MHz, CDCl₃) δ 7.75 (d, J = 7.3 Hz, 2H), 7.54 (s, 1H), 7.45 (d, J = 8.5 Hz, 2H), 7.41 (t, J = 7.5 Hz, 2H), 7.33 (t, J = 7.0 Hz, 1H), 7.28 (d, J = 8.5 Hz, 2H), 5.48 (t, J = 8.9 Hz, 1H), 5.11 (dd, J = 14.3, 7.2 Hz, 1H), 4.91 (dd, J = 14.2, 8.6 Hz, 1H); **¹³C NMR** (150 MHz, CDCl₃) δ 138.6, 132.4, 129.2, 128.9, 128.4, 125.8, 122.9, 119.9, 58.4, 25.4; **HRMS (ESI)**: calcd For C₁₆H₁₄BrIN₃⁺ (M+H)⁺: 453.9410; Found: 453.9408.

1-(2-iodo-2-(m-tolyl) ethyl)-4-phenyl-1H-1,2,3-triazole (7f)



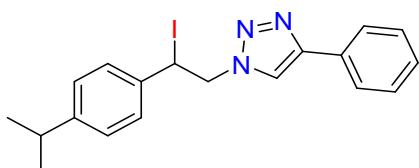
White solid. Mp: 133-135 °C. **¹H NMR** (600 MHz, CDCl₃) δ 7.73 (d, J = 7.74 Hz, 2H), 7.49 (s, 1H), 7.40 (t, J = 7.7 Hz, 2H), 7.32 (t, J = 7.4 Hz, 1H), 7.22-7.19 (m, 3H), 7.12-7.08 (m, 1H), 5.47 (t, J = 7.9 Hz, 1H), 5.14 (dd, J = 14.3, 7.4 Hz, 1H), 4.91 (dd, J = 14.3, 8.3 Hz, 1H), 2.32 (s, 3H); **¹³C NMR** (150 MHz, CDCl₃) δ 147.3, 139.4, 139.9, 130.3, 129.9, 129.0, 128.8, 128.3, 128.2, 125.7, 124.6, 120.1, 58.6, 27.2, 21.3; **HRMS (ESI)**: calcd For C₁₇H₁₇IN₃⁺ (M+H)⁺: 390.0462; Found: 390.0458.

1-(2-iodo-2-(o-tolyl) ethyl)-4-phenyl-1H-1,2,3-triazole (7g)



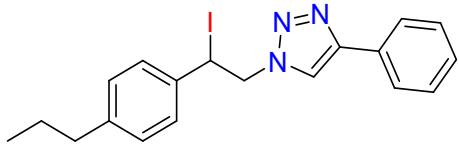
White solid. Mp:135-137 °C. **¹H NMR** (600 MHz, CDCl₃) δ 7.74 (d, *J* = 7.6 Hz, 2H), 7.60 (d, *J* = 7.8 Hz, 1H), 7.54 (s, 1H), 7.40 (t, *J* = 7.6 Hz, 2H), 7.33-7.28 (m, 2H), 7.19 (t, *J* = 7.5 Hz, 1H), 7.08 (d, *J* = 7.6 Hz, 1H), 5.75 (t, *J* = 7.7 Hz, 1H), 5.21 (dd, *J* = 14.2, 7.4 Hz, 1H), 4.99 (dd, *J* = 14.3, 8.1 Hz, 1H), 2.21 (s, 3H); **¹³C NMR** (150 MHz, CDCl₃) δ 147.3, 137.6, 135.5, 131.2, 130.3, 128.9, 128.8, 128.2, 127.1, 127.1, 125.7, 120.1, 58.0, 26.9, 23.6, 19.1; **HRMS (ESI)**: calcd For C₁₇H₁₇IN₃⁺ (M+H)⁺ : 390.0462; Found: 390.0465.

1-(2-iodo-2-(4-isopropylphenyl) ethyl)-4-phenyl-1H-1,2,3-triazole (7h)



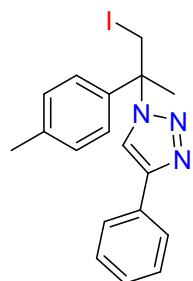
White solid. Mp:134-136 °C. **¹H NMR** (600 MHz, CDCl₃) δ 7.72 (d, *J* = 7.3 Hz, 2H), 7.46 (s, 1H), 7.39 (t, *J* = 7.6 Hz, 2H), 7.32 (d, *J* = 7.9 Hz, 3H), 7.17 (d, *J* = 8.1 Hz, 2H), 5.49 (t, *J* = 7.8 Hz, 1H), 5.14 (dd, *J* = 14.3, 7.4 Hz, 1H), 4.89 (dd, *J* = 14.3, 8.3 Hz, 1H), 2.91-2.86 (m, 1H), 1.23 (d, *J* = 6.9 Hz, 6H); **¹³C NMR** (150 MHz, CDCl₃) δ 150.0, 147.3, 136.9, 130.3, 128.8, 128.2, 127.6, 127.2, 125.7, 120.1, 58.7, 33.8, 27.4, 23.8, 23.7; **HRMS (ESI)**: calcd For C₁₉H₂₁IN₃⁺ (M+H)⁺ : 418.0775; Found: 418.0774.

1-(2-iodo-2-(4-propylphenyl) ethyl)-4-phenyl-1H-1,2,3-triazole(7i)



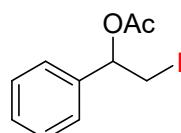
White solid. Mp:128-130 °C. **¹H NMR** (600 MHz, CDCl₃) δ 7.71 (d, J = 7.9 Hz, 2H), 7.44 (s, 1H), 7.39 (t, J = 7.6 Hz, 2H), 7.32-7.30 (m, 3H), 7.12 (d, J = 7.9 Hz, 2H), 5.49 (t, J = 7.9 Hz, 1H), 5.15 (dd, J = 14.2, 7.3 Hz, 1H), 4.90 (dd, J = 14.2, 8.5 Hz, 1H), 2.55 (t, J = 7.5 Hz, 2H), 1.64-1.59 (m, 2H), 0.92 (t, J = 7.3 Hz, 3H); **¹³C NMR** (150 MHz, CDCl₃) δ 147.3, 143.9, 136.8, 130.3, 129.2, 128.8, 128.2, 127.5, 125.7, 120.0, 58.7, 37.7, 27.3, 24.2, 13.7; **HRMS (ESI)**: calcd For C₁₉H₂₁IN₃⁺ (M+H)⁺ : 418.0775; Found: 418.0774.

1-(1-iodo-2-(p-tolyl) propan-2-yl)-4-phenyl-1H-1,2,3-triazole (7j)



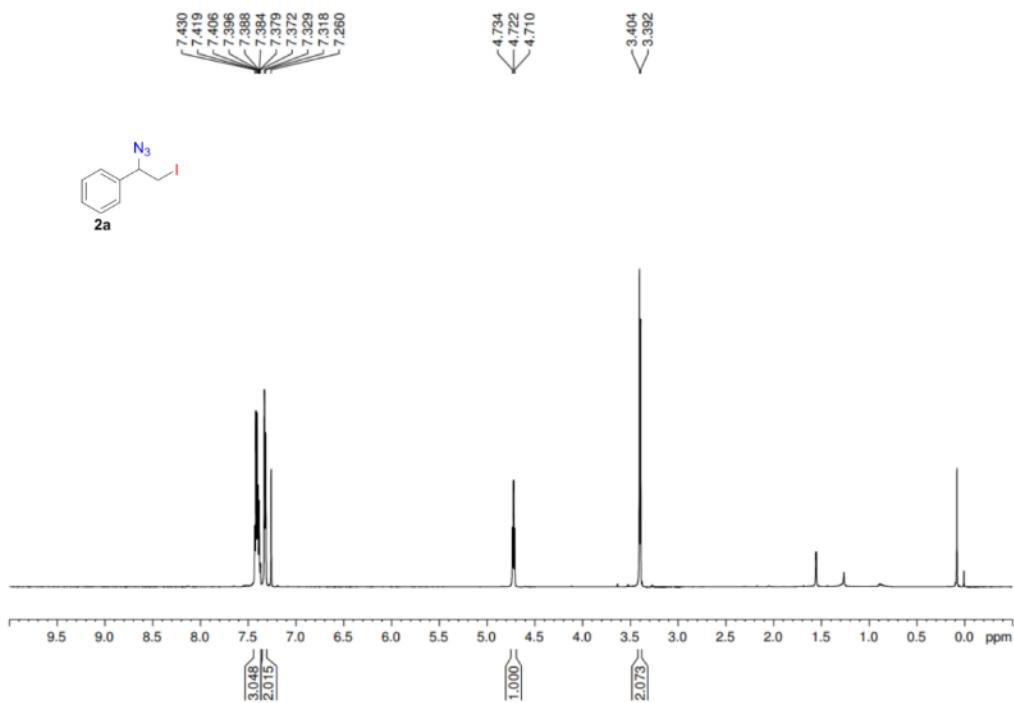
White solid. Mp:134-136 °C. **¹H NMR** (600 MHz, CDCl₃) δ 7.84 (d, J = 7.3 Hz, 2H), 7.69 (s, 1H), 7.41 (t, J = 7.6 Hz, 2H), 7.32 (t, J = 7.4 Hz, 1H), 7.16 (d, J = 8.2 Hz, 2H), 7.03 (d, J = 8.3 Hz, 2H), 4.39 (d, J = 10.8 Hz, 1H), 4.02 (d, J = 10.7 Hz, 1H), 2.35 (s, 3H), 2.22 (s, 3H); **¹³C NMR** (150 MHz, CDCl₃) δ 147.5, 138.6, 138.1, 130.5, 129.6, 128.8, 128.2, 125.7, 125.4, 119.1, 65.8, 28.5, 21.0, 15.6; **HRMS (ESI)**: calcd For C₁₈H₁₉IN₃⁺ (M+H)⁺ : 404.0618; Found: 404.0621.

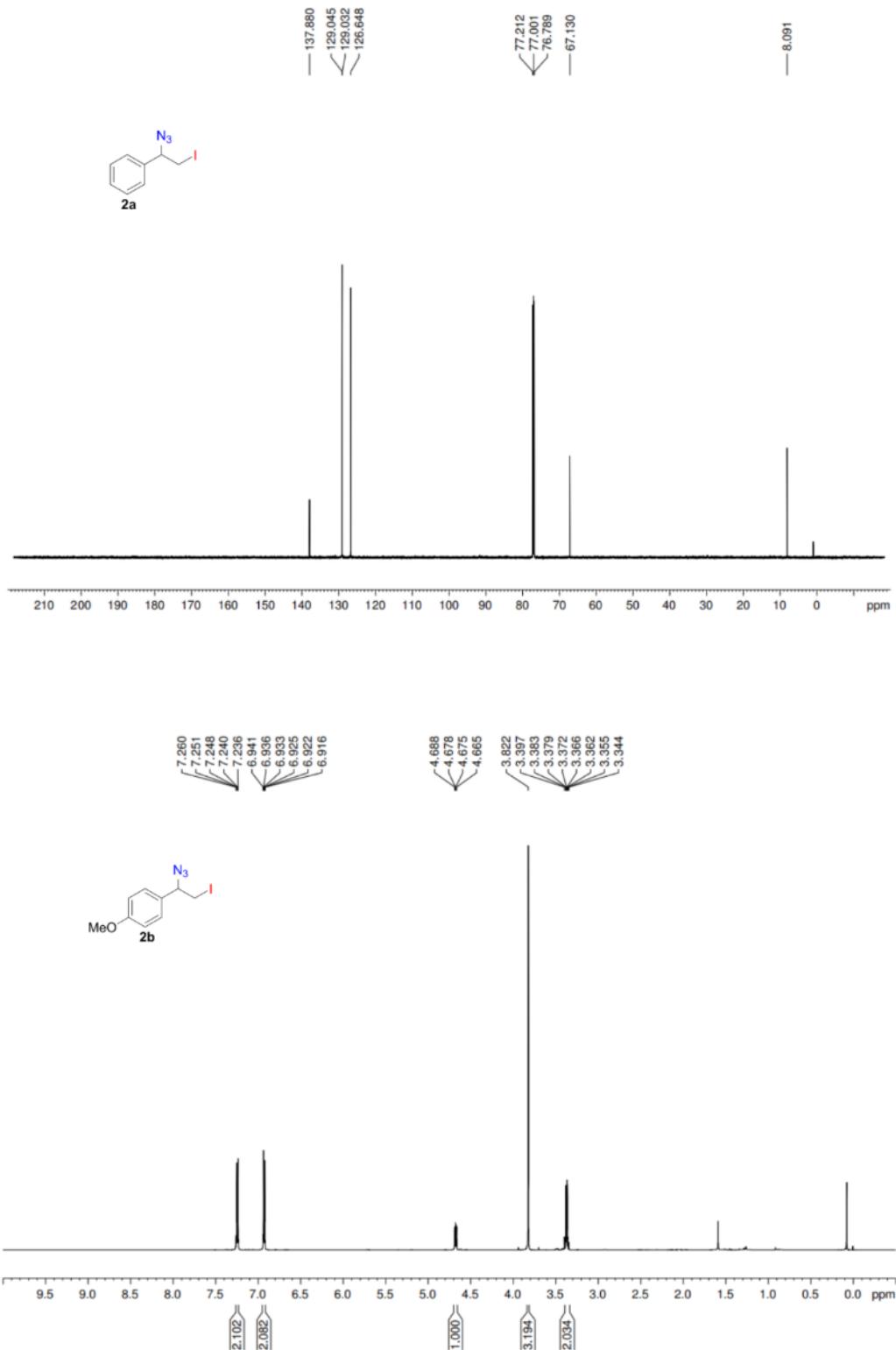
2-iodo-1-phenylethyl acetate (8)¹

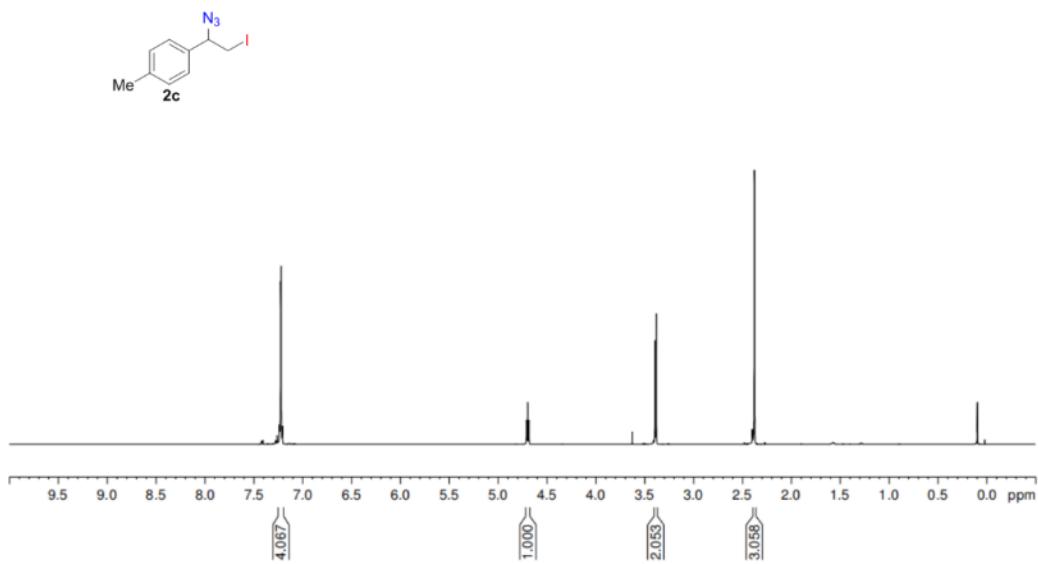
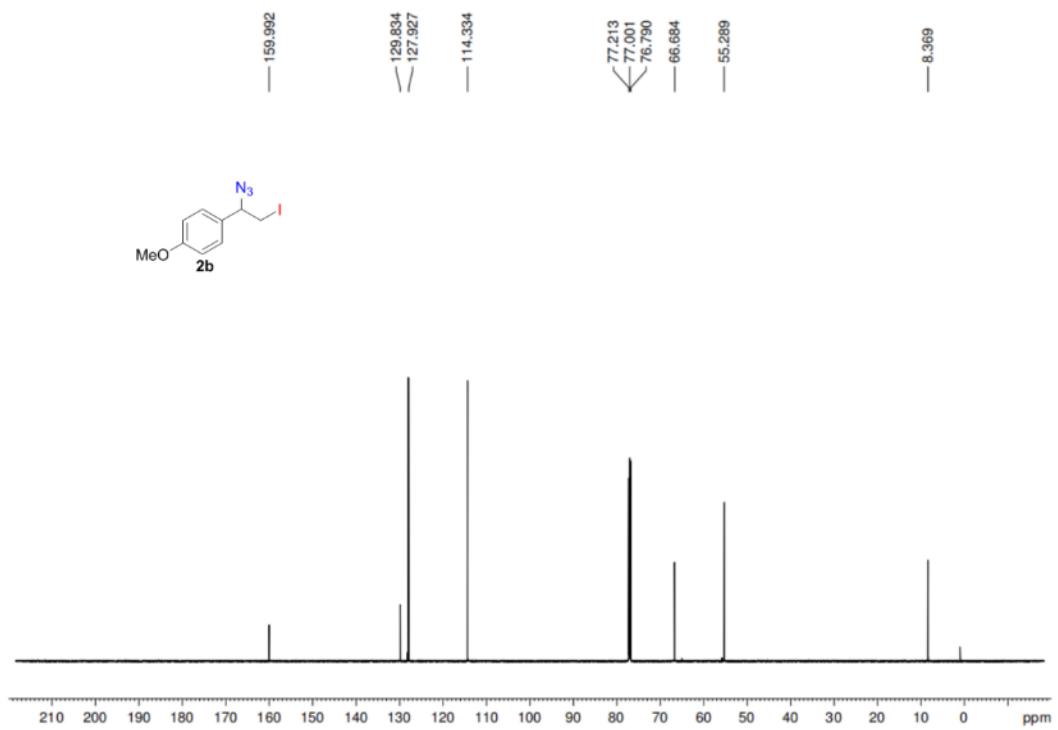


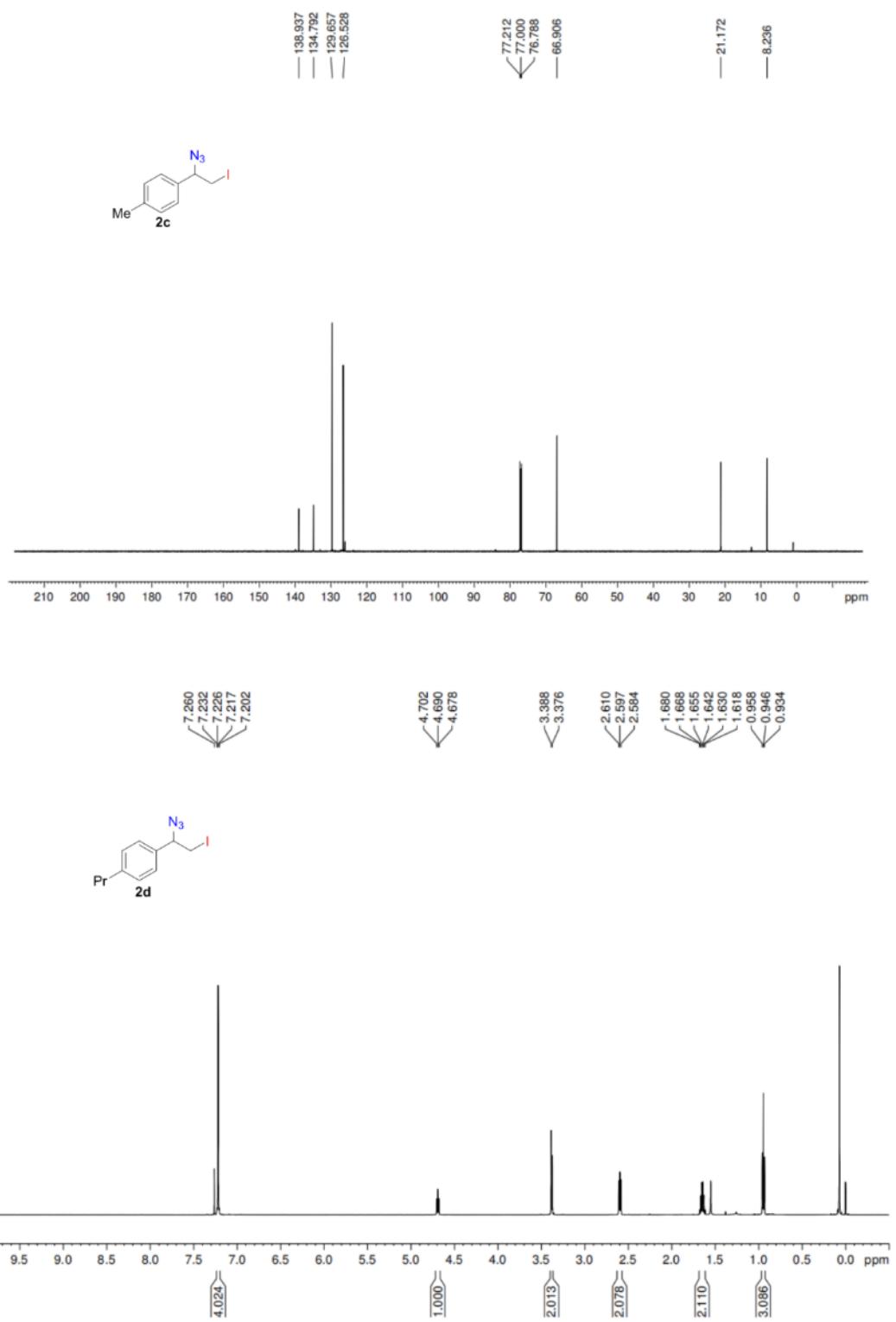
Colorless oil. **¹H NMR** (600 MHz, CDCl₃) δ 7.37-7.34 (m, 5H), 5.88 (dd, J = 7.8, 5.4 Hz, 1H), 3.50-3.45 (m, 2H), 2.14 (s, 3H). **¹³C NMR** (150 MHz, CDCl₃) δ 169.7, 138.4, 128.7, 128.7, 126.4, 75.1, 21.0, 7.7.

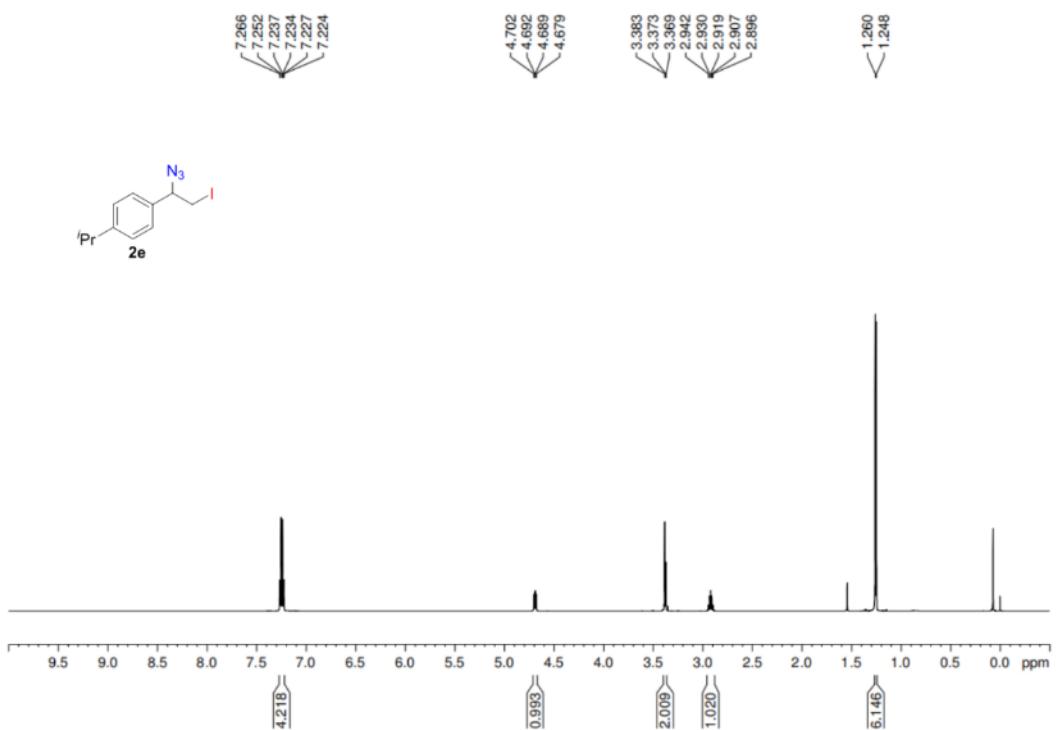
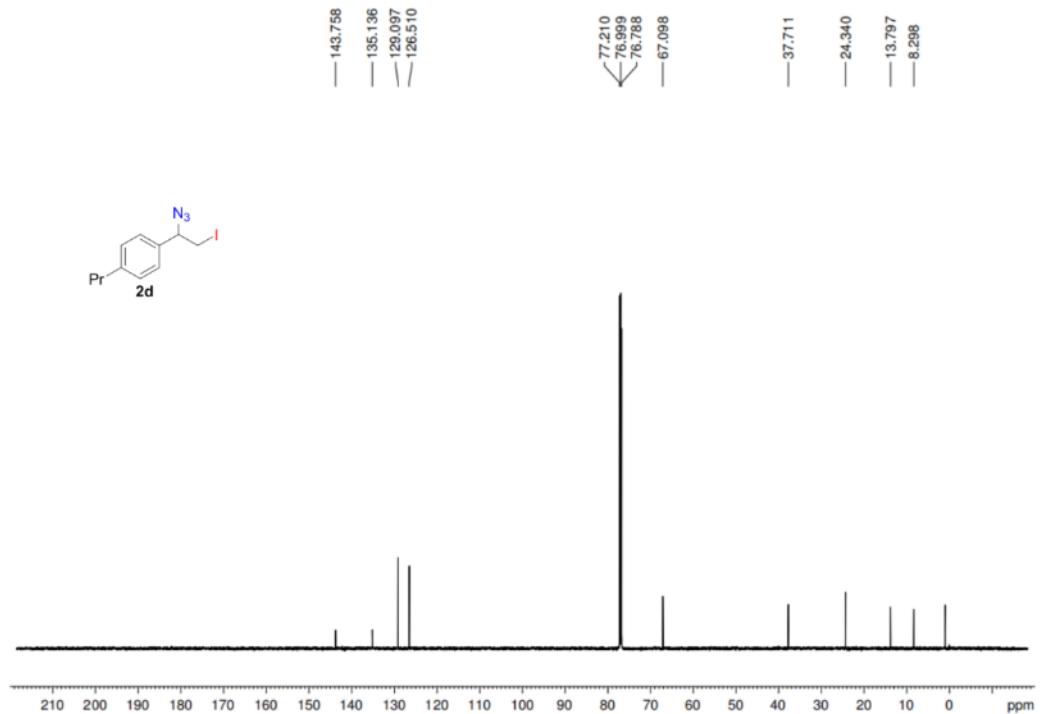
8. ^1H NMR and ^{13}C NMR spectra of new azosulfones and all products

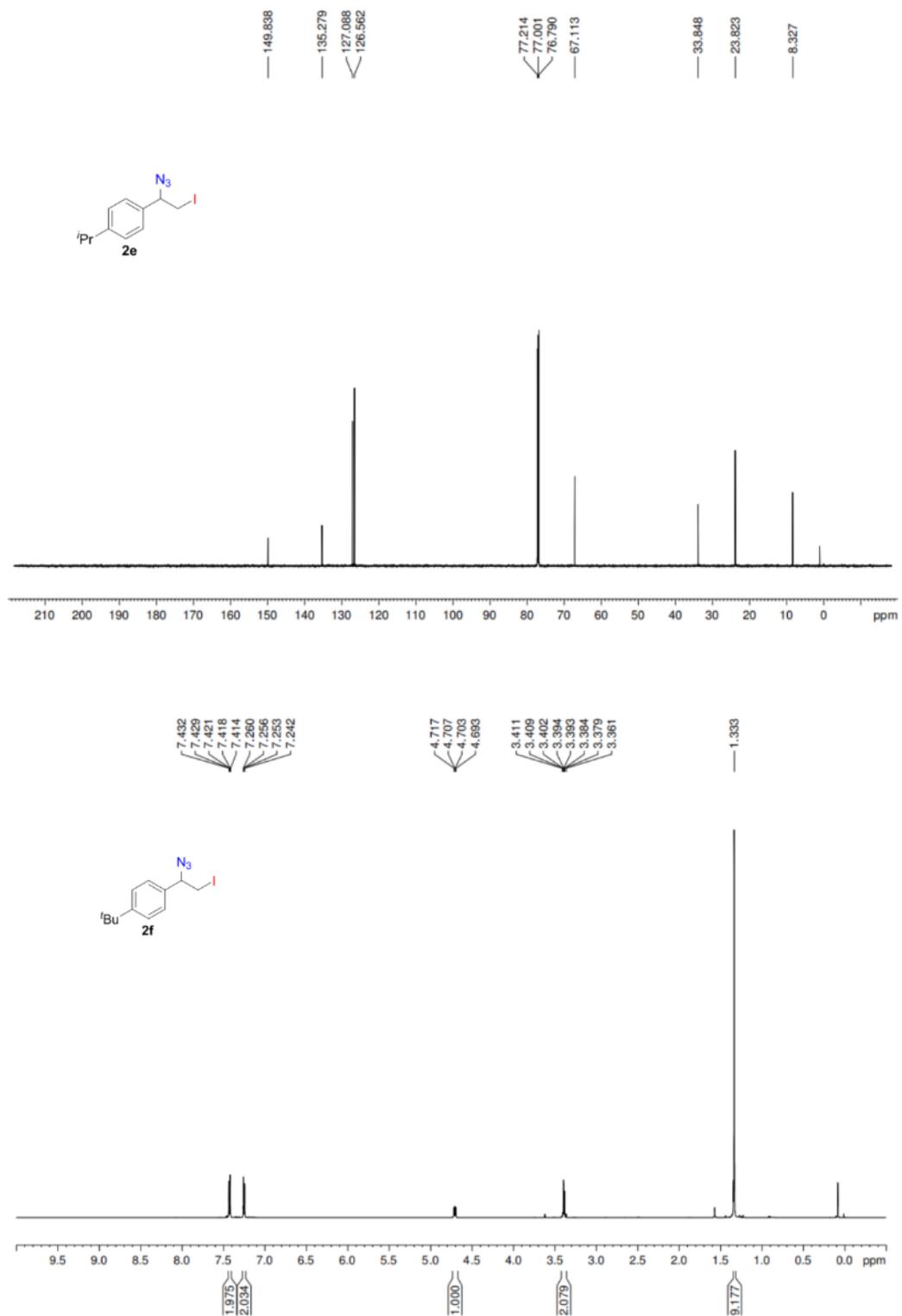


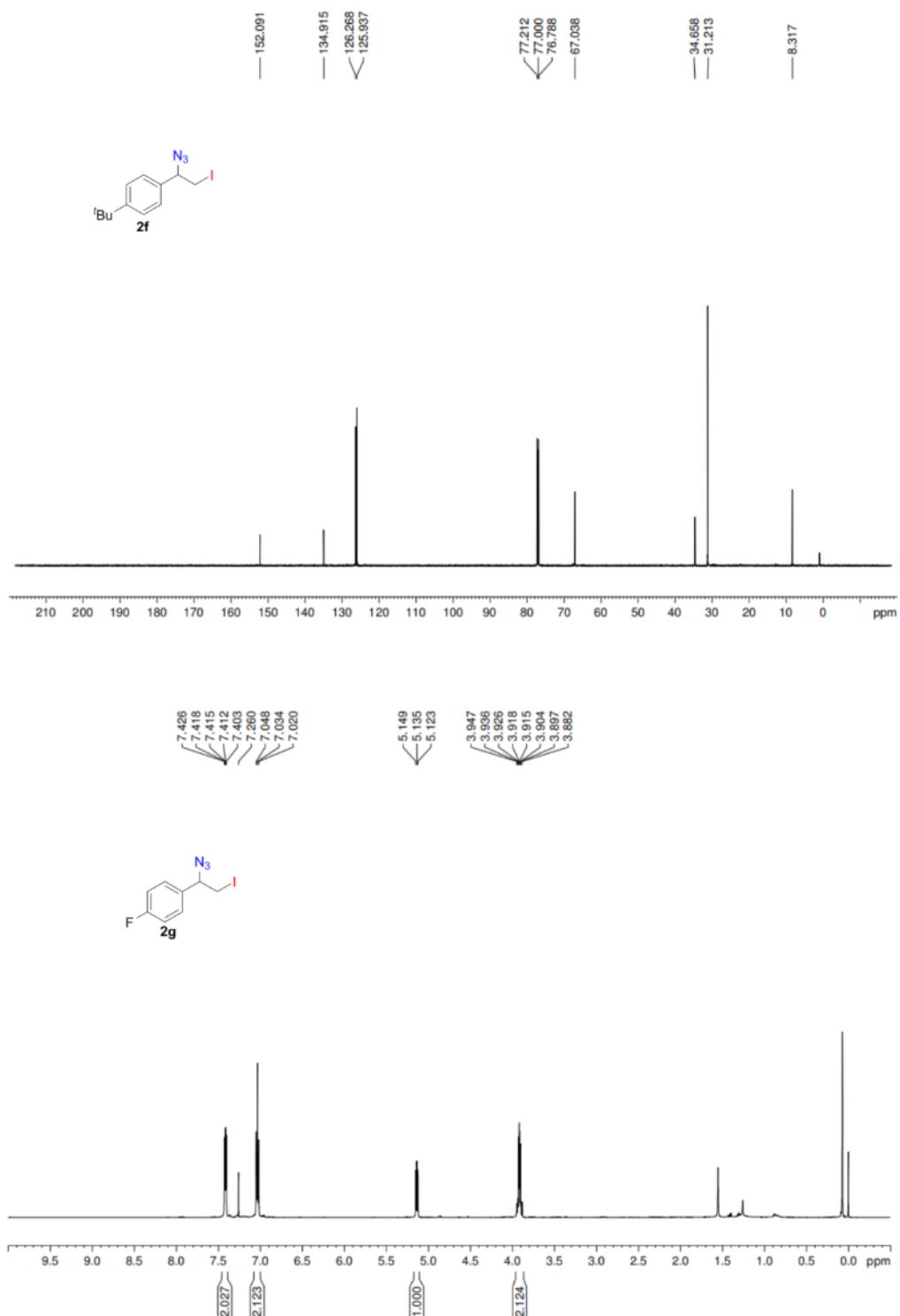


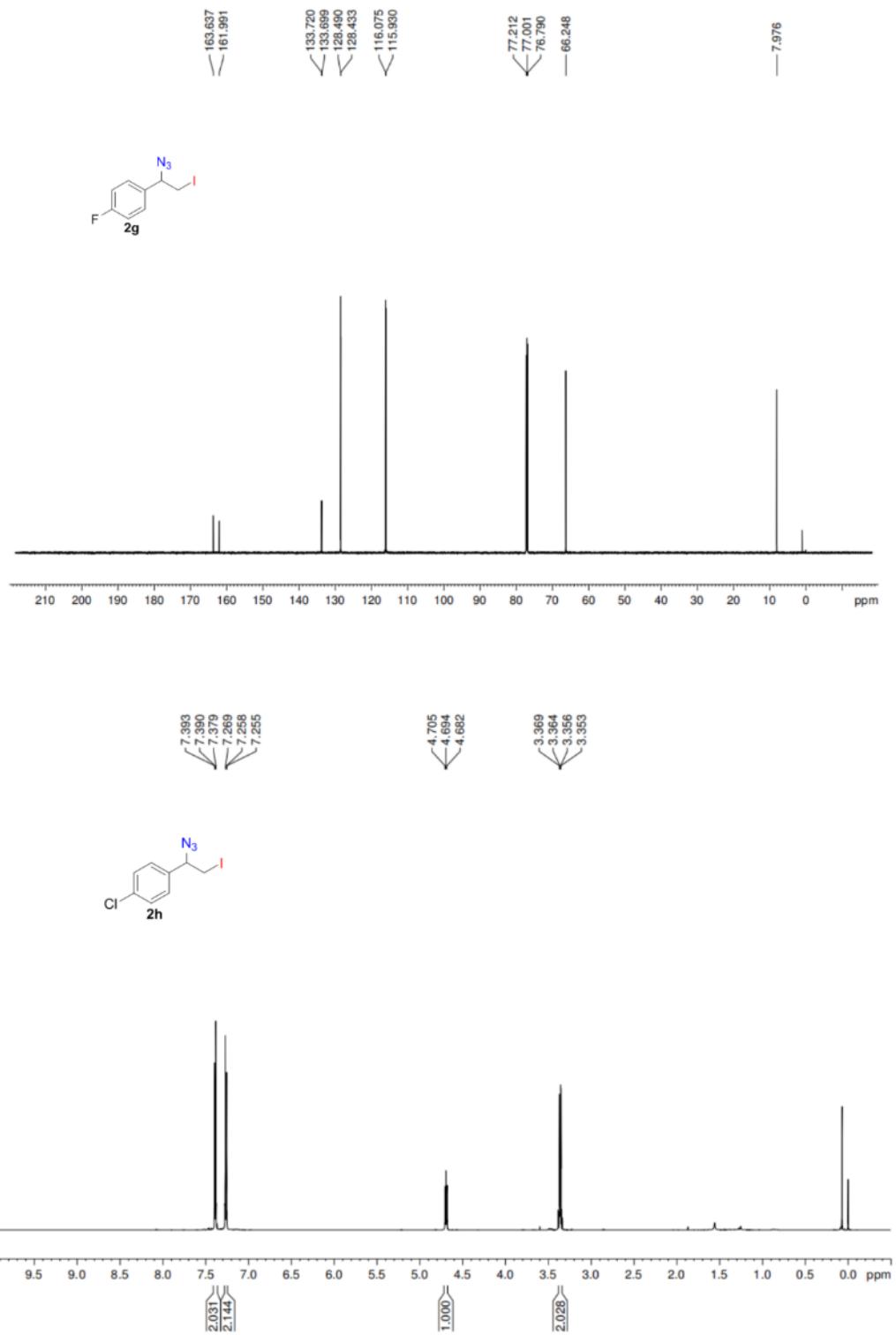


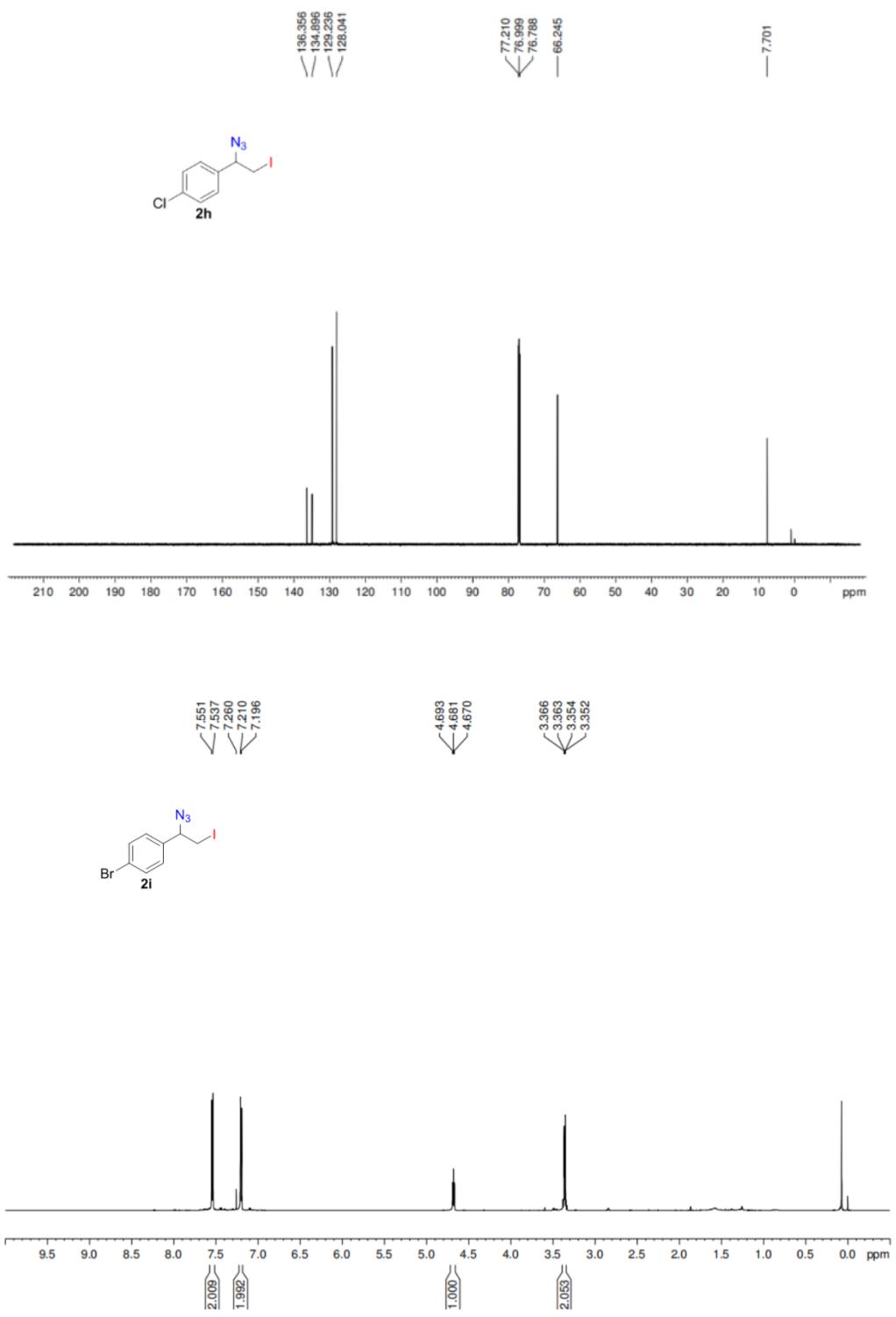


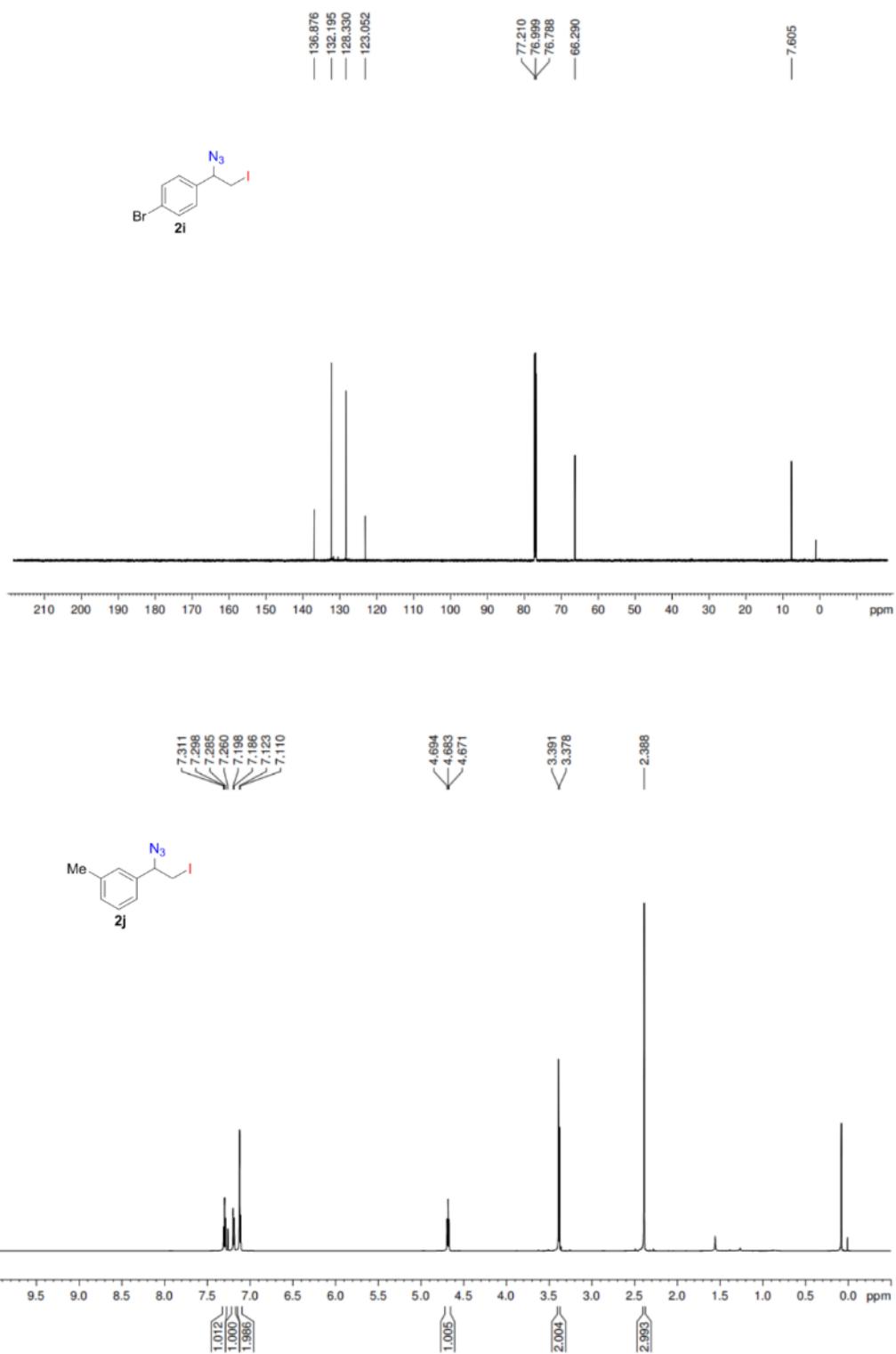


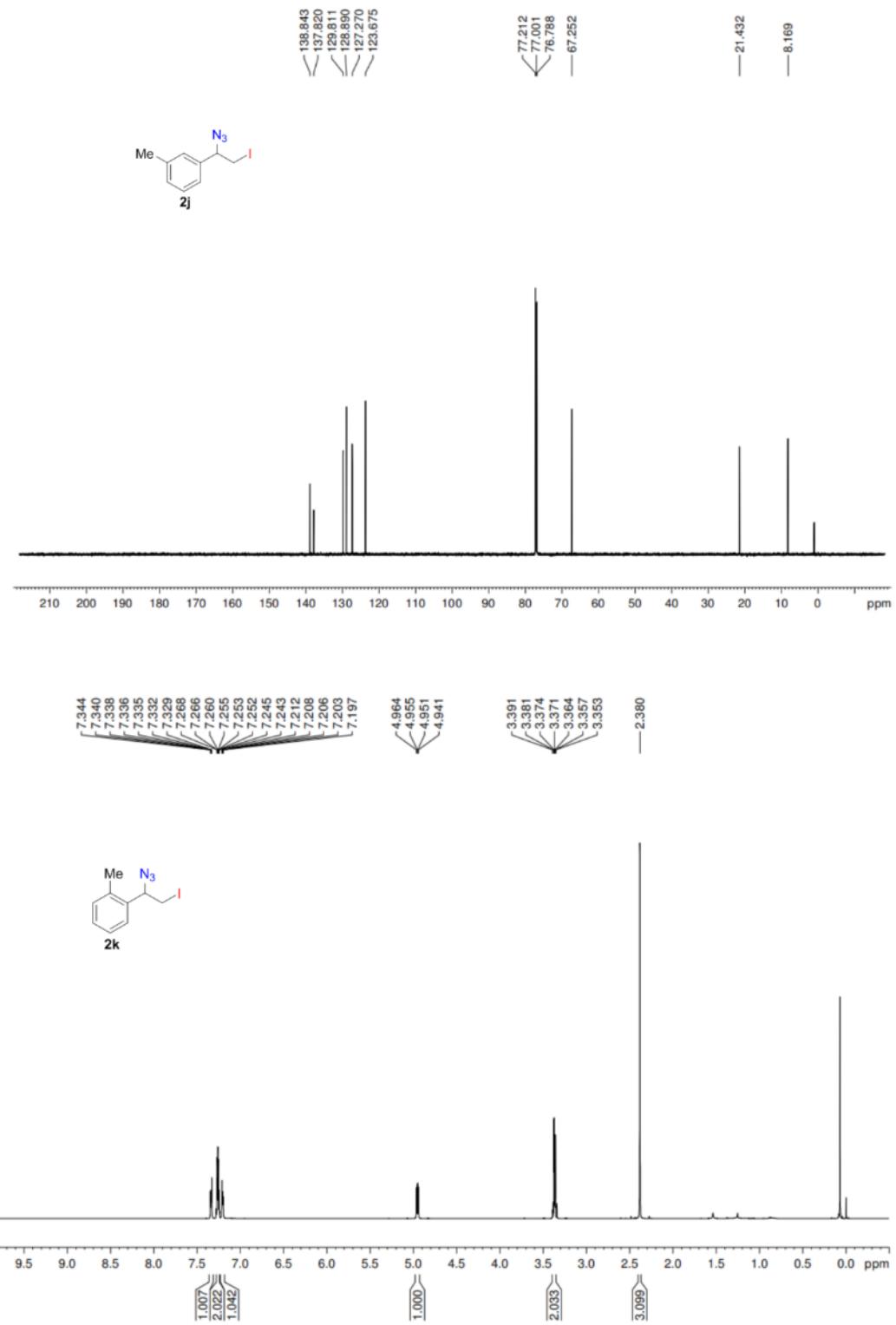


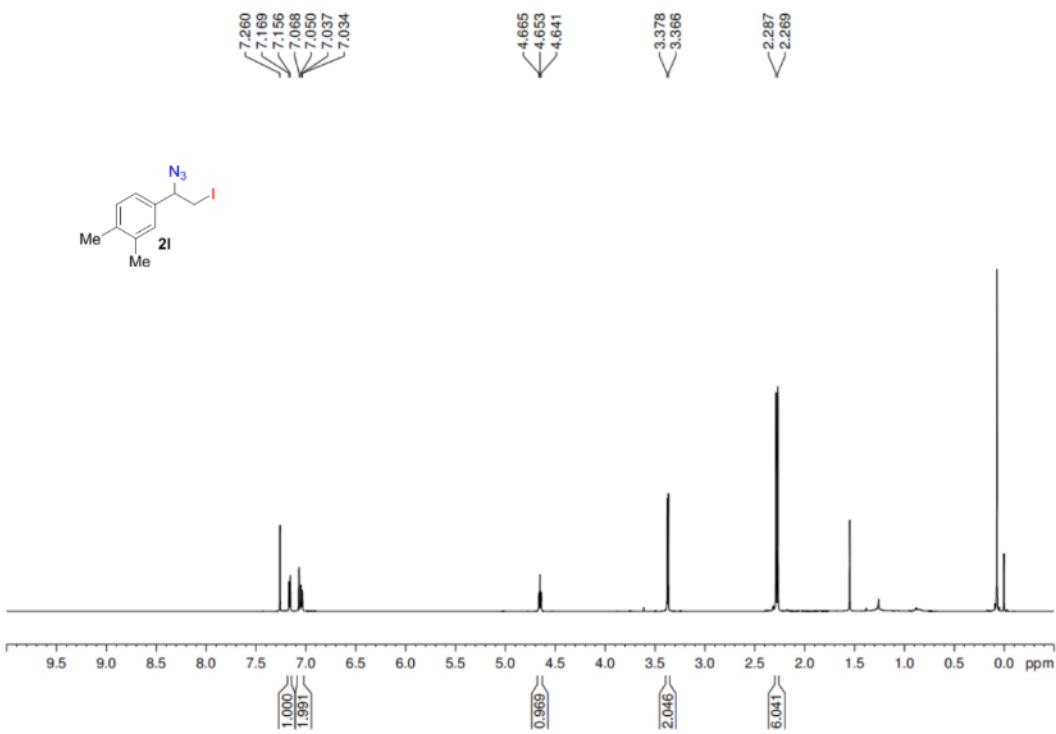
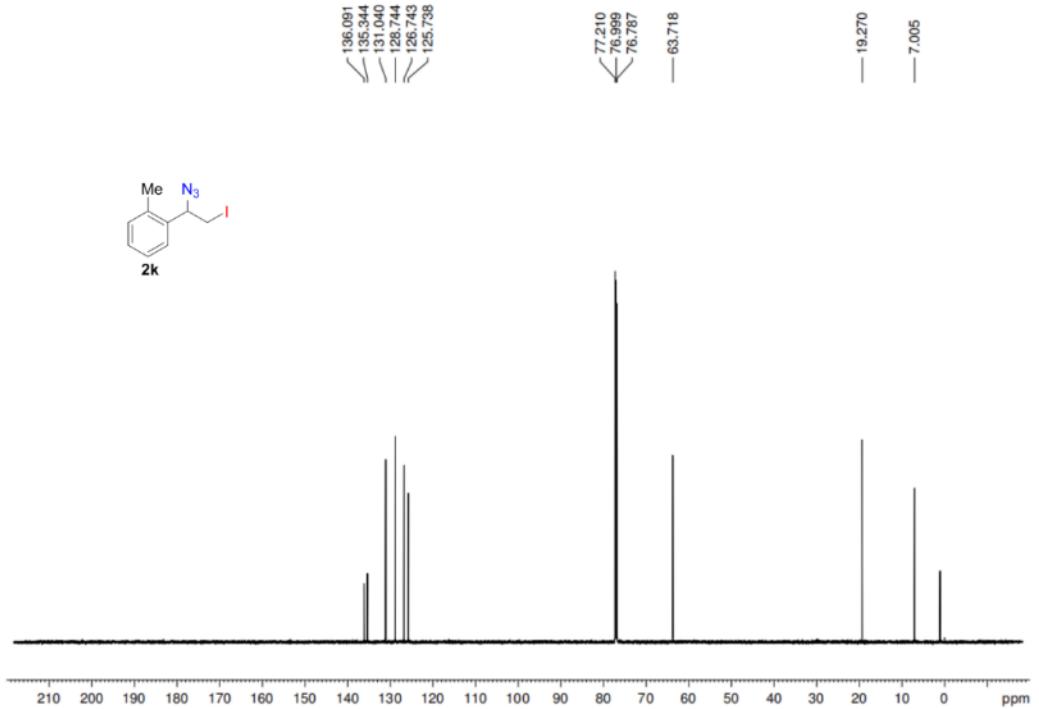


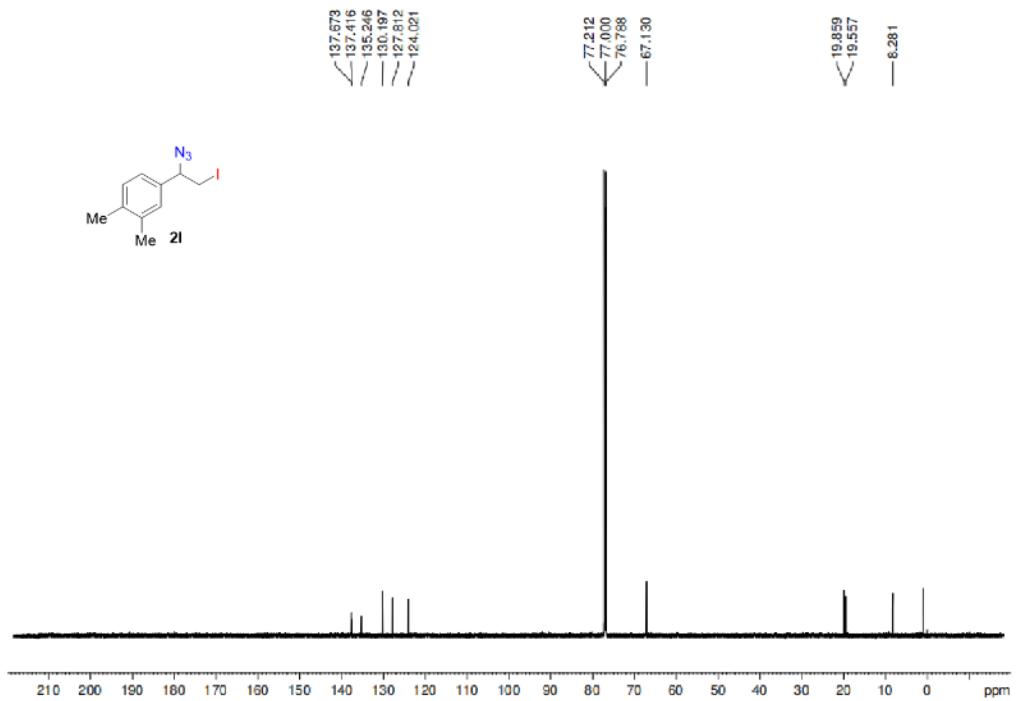


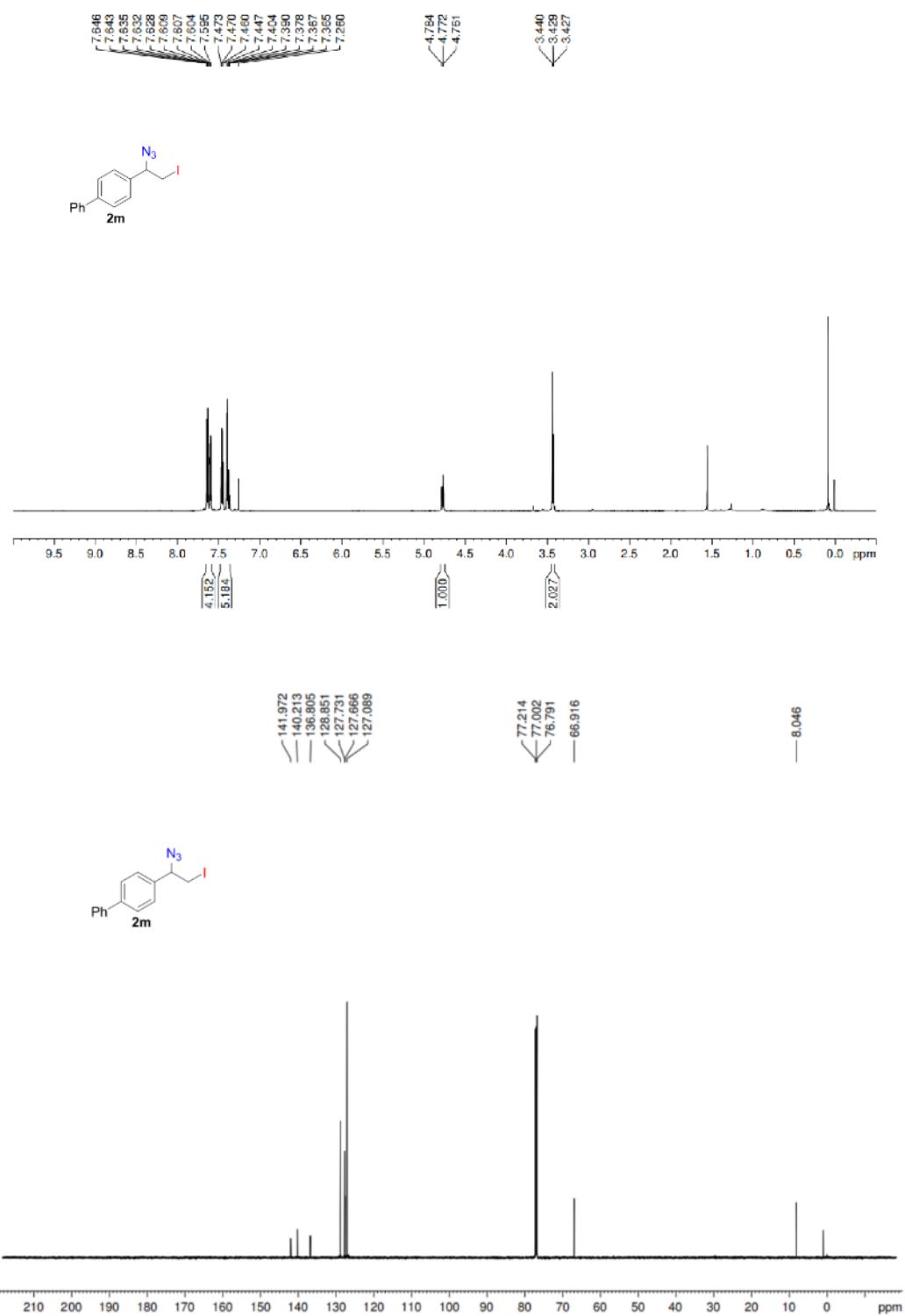


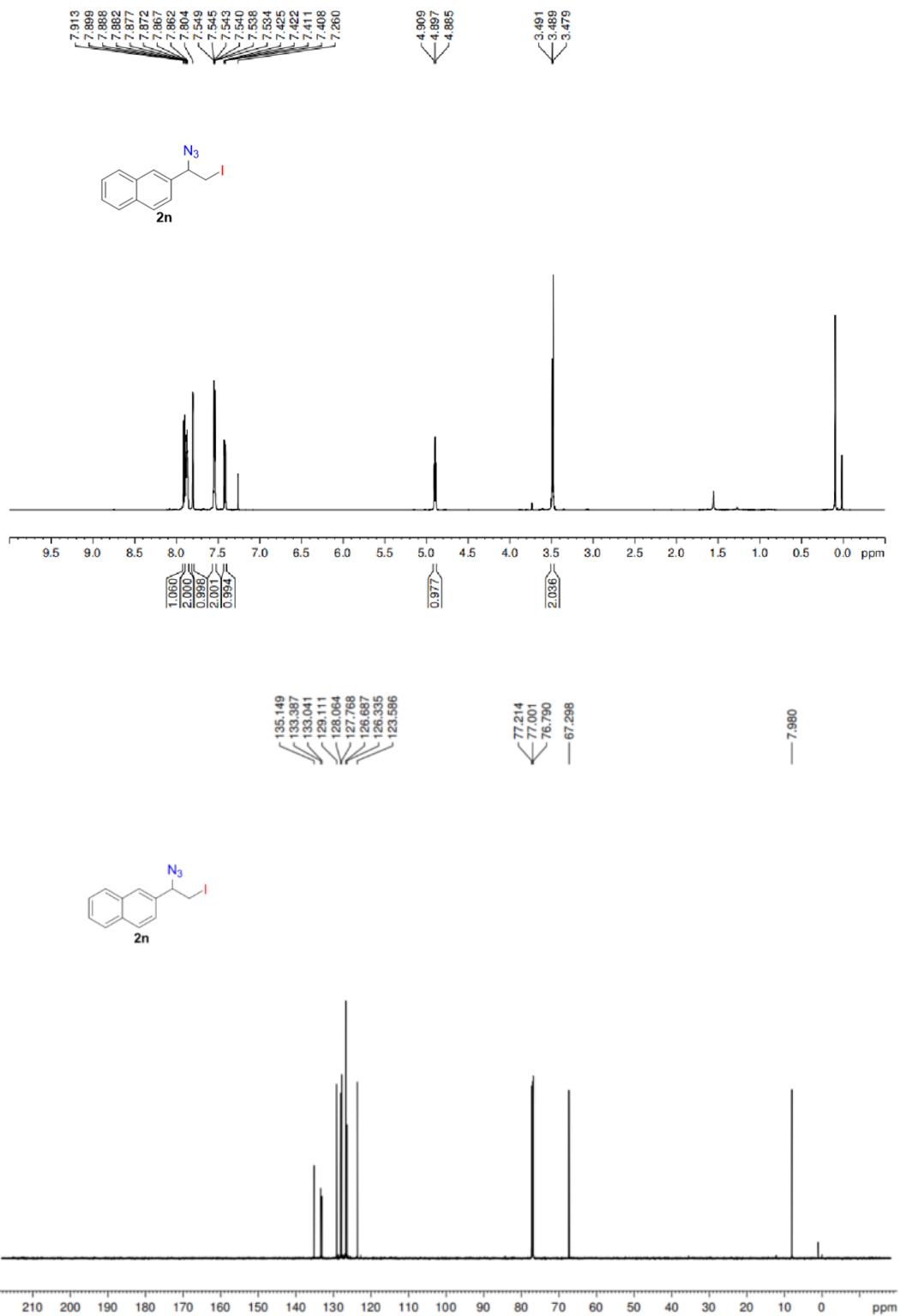


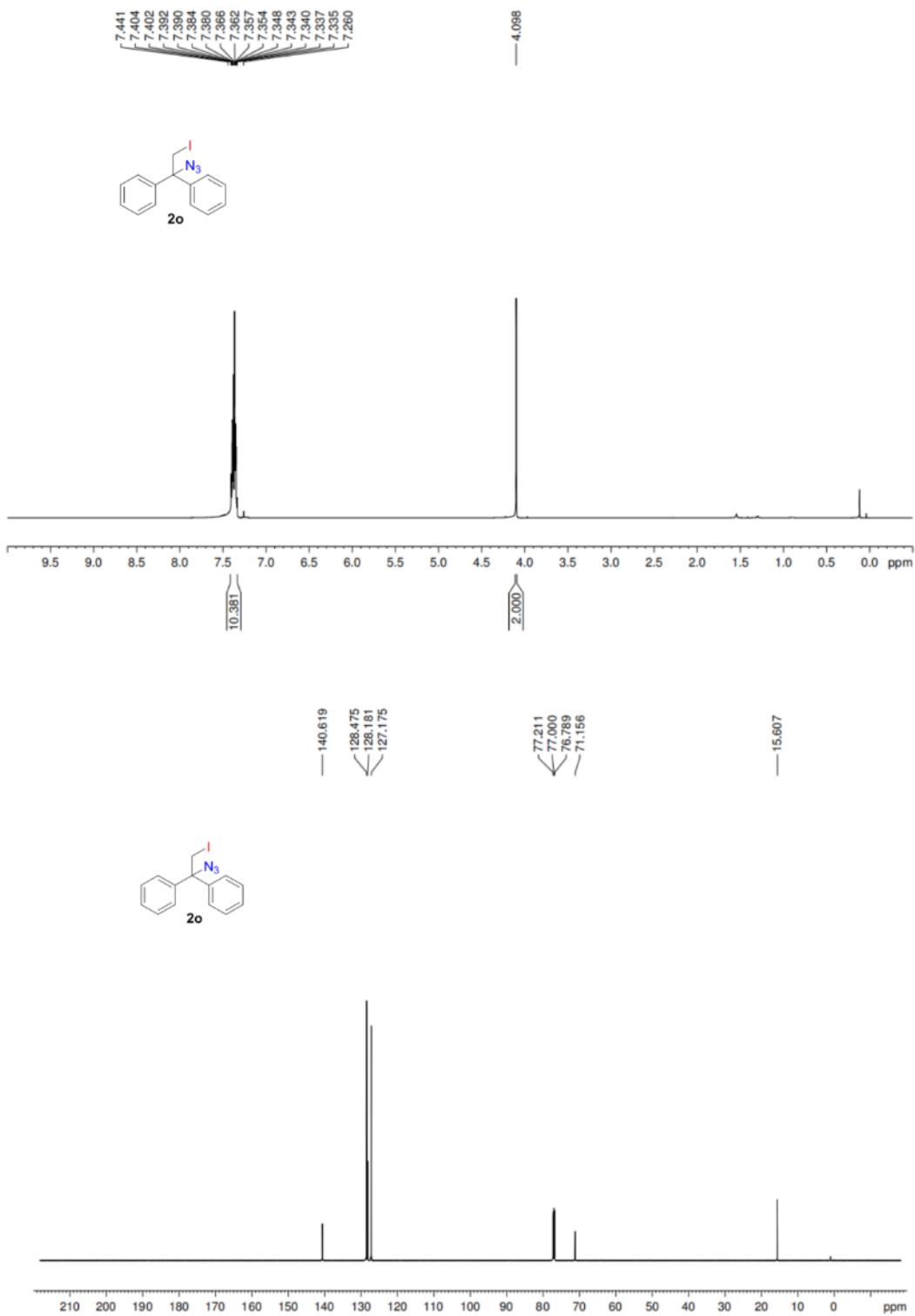


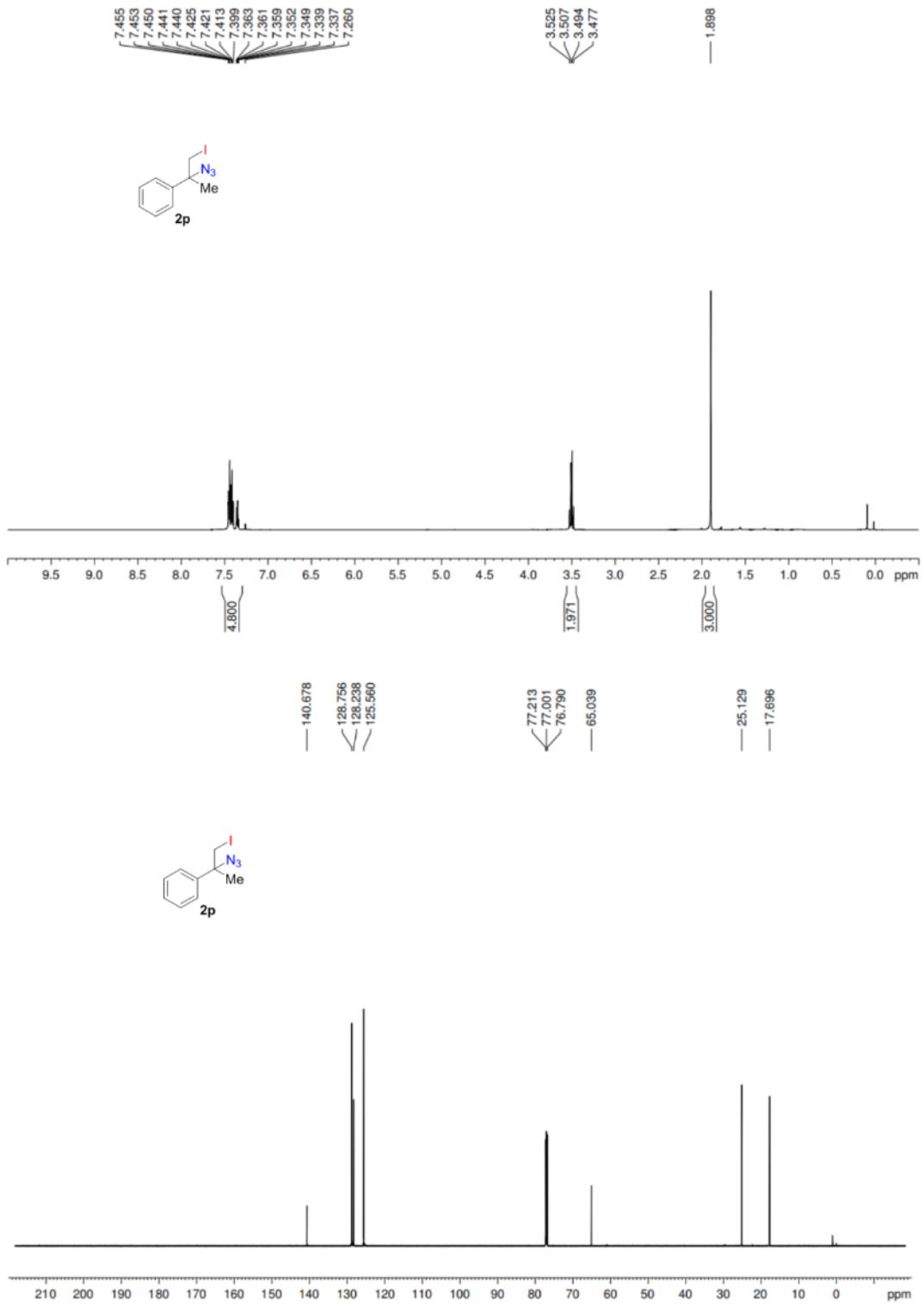


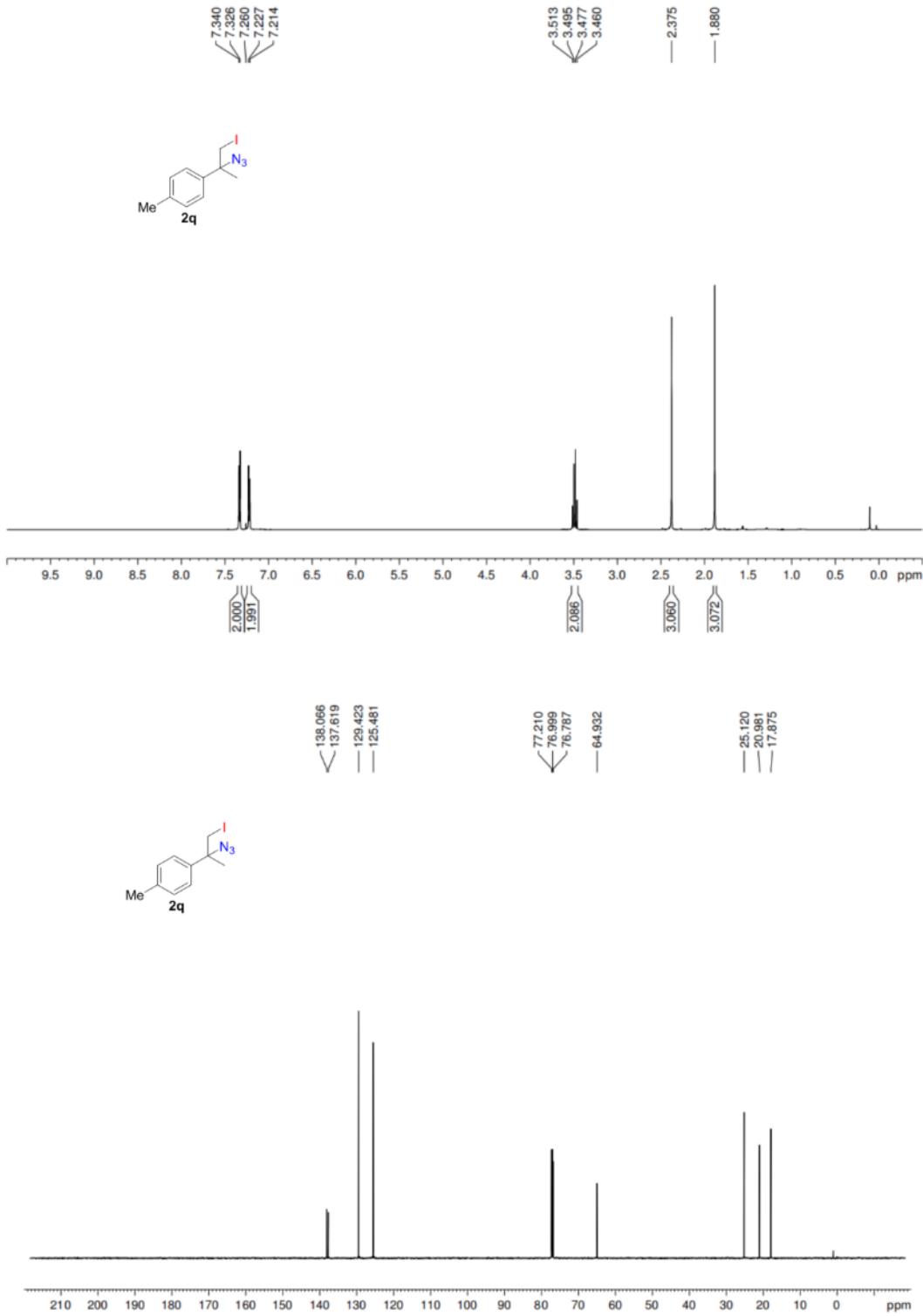


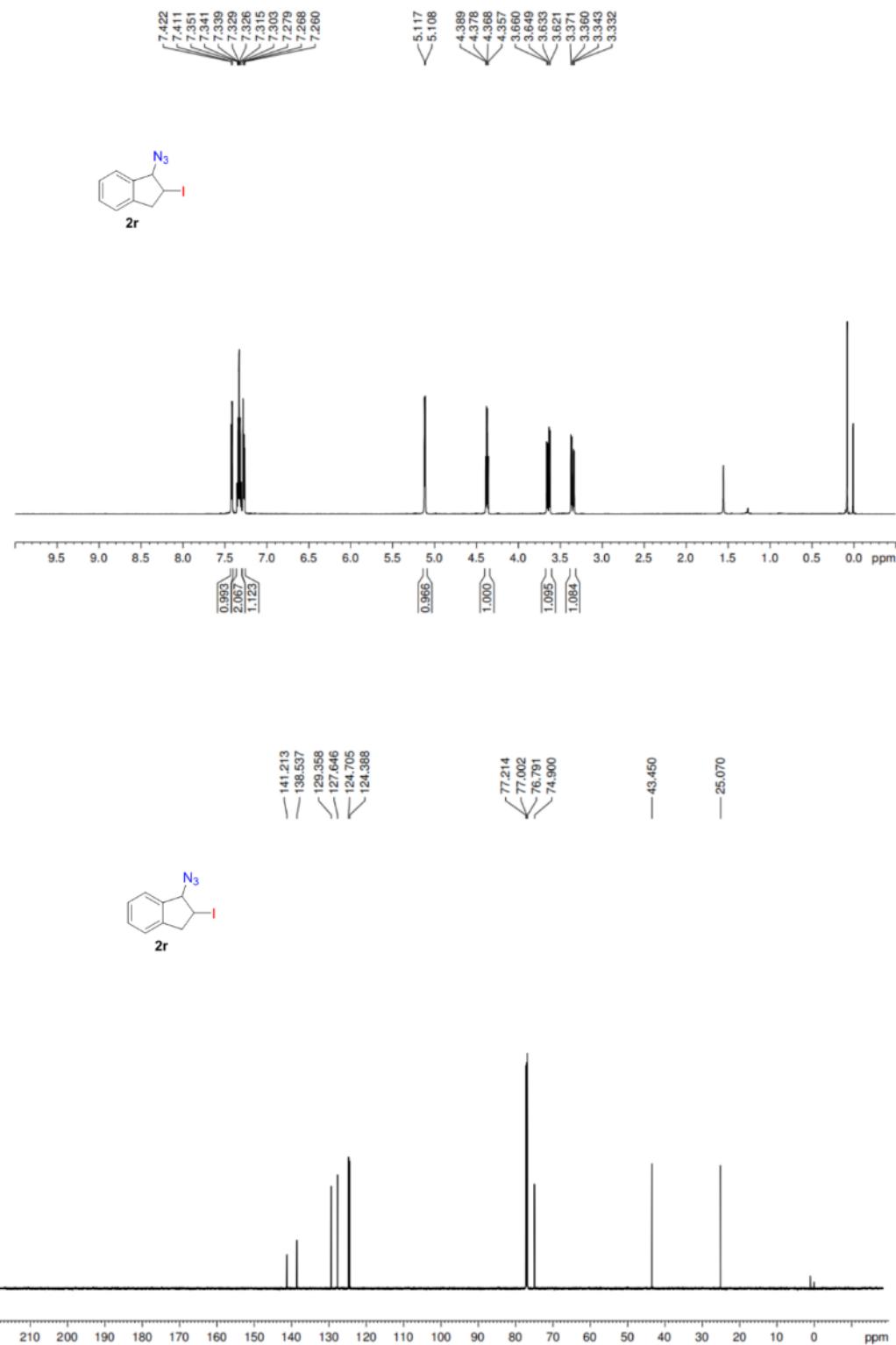


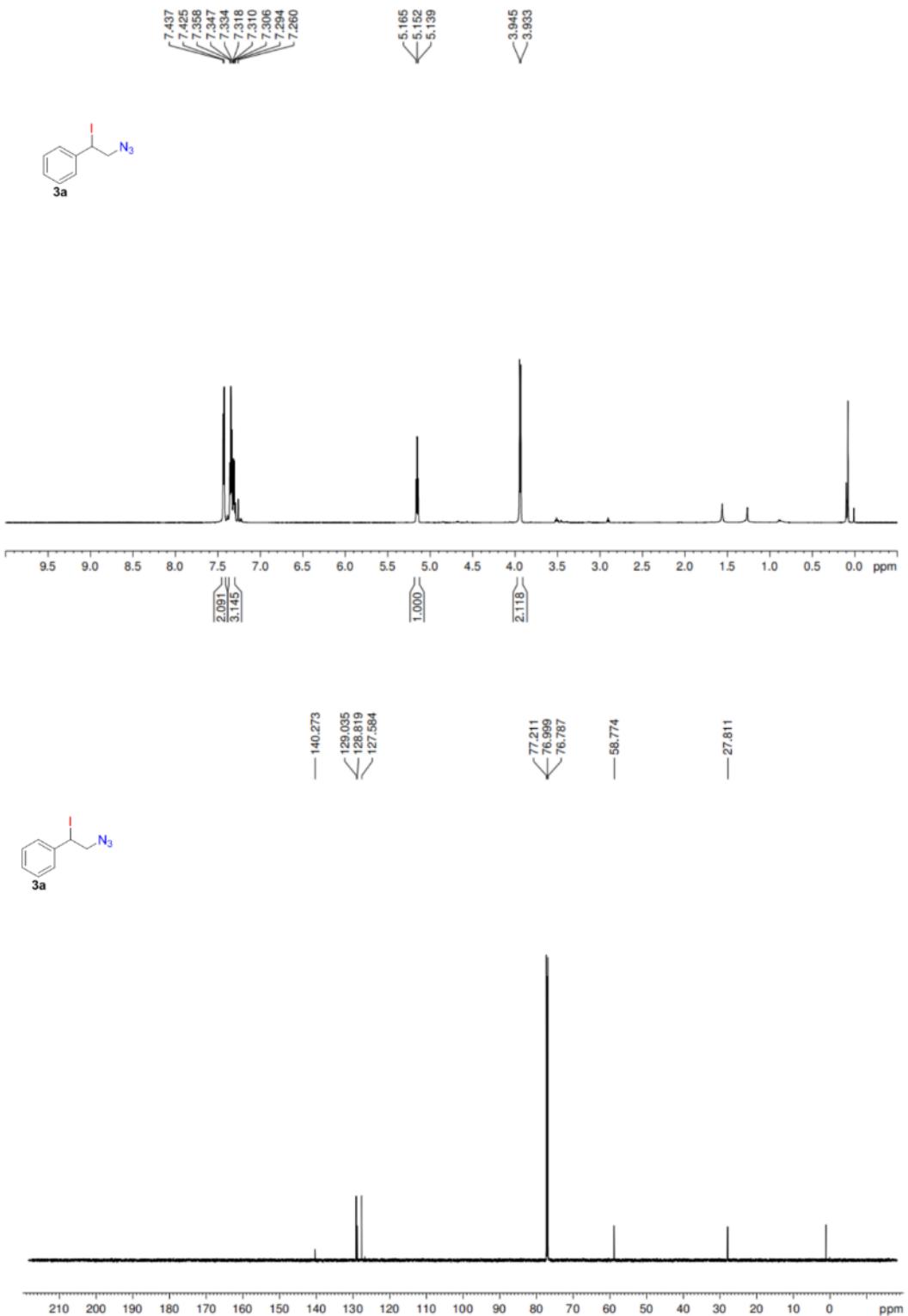


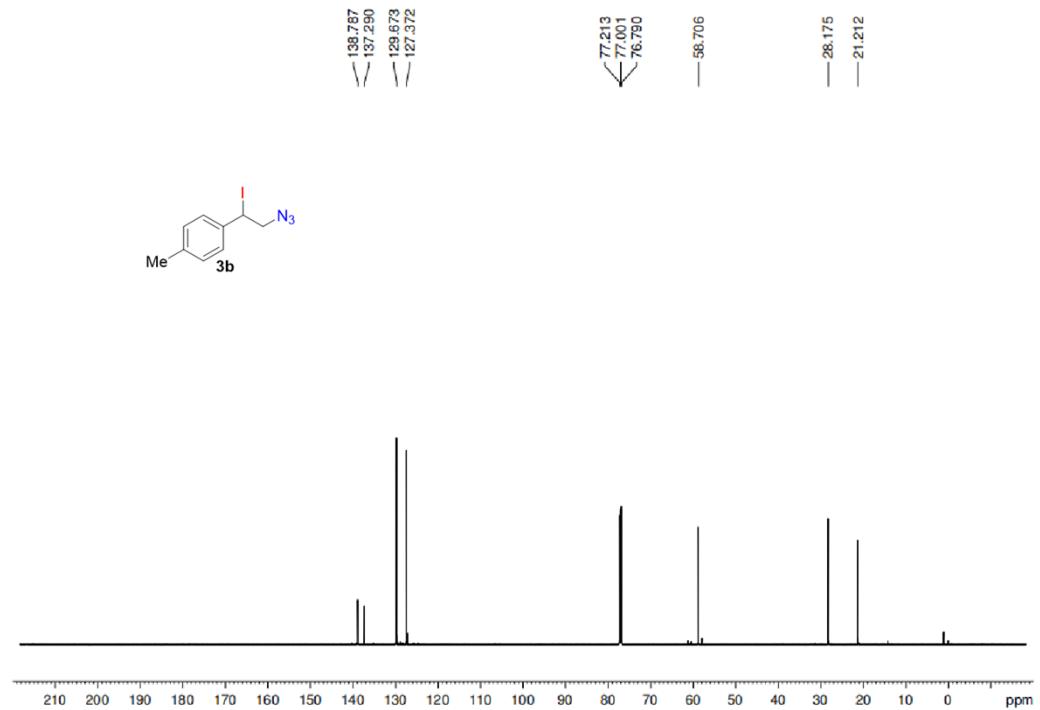
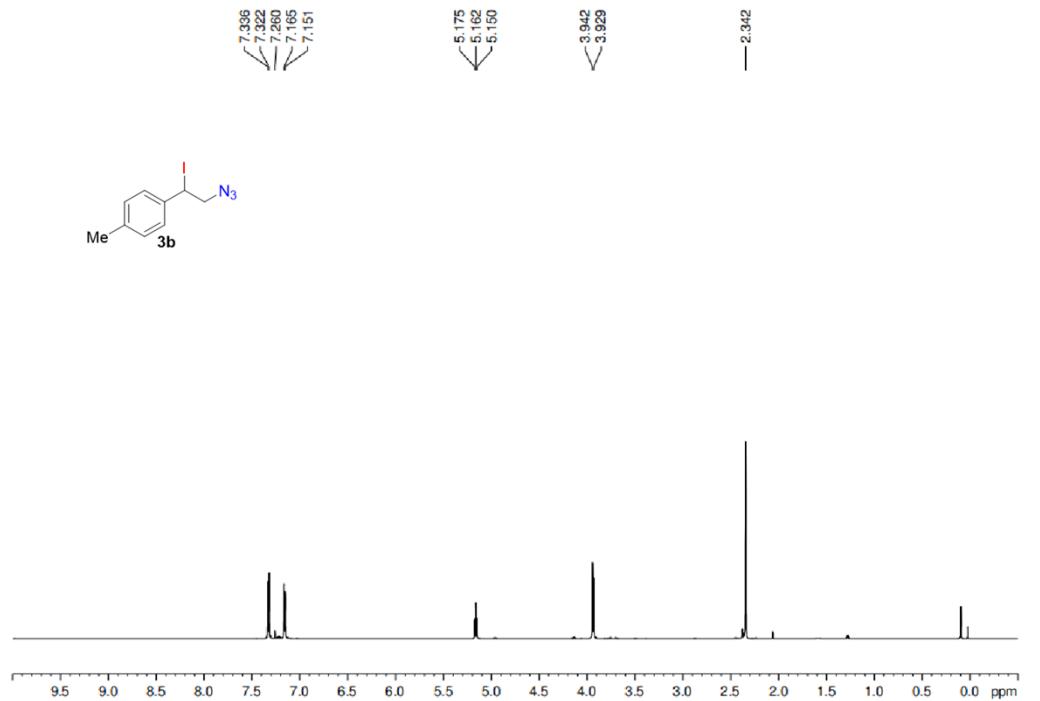


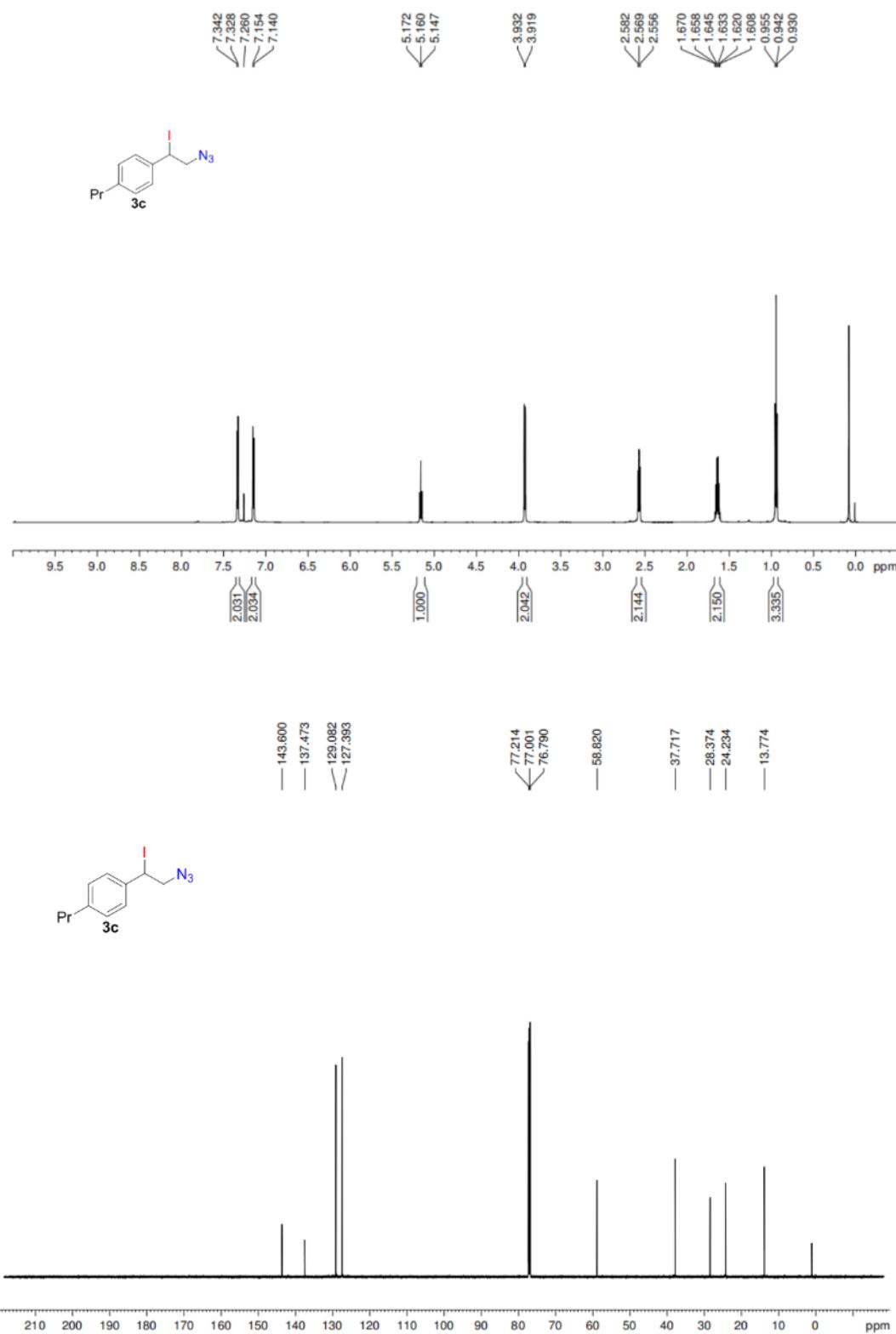


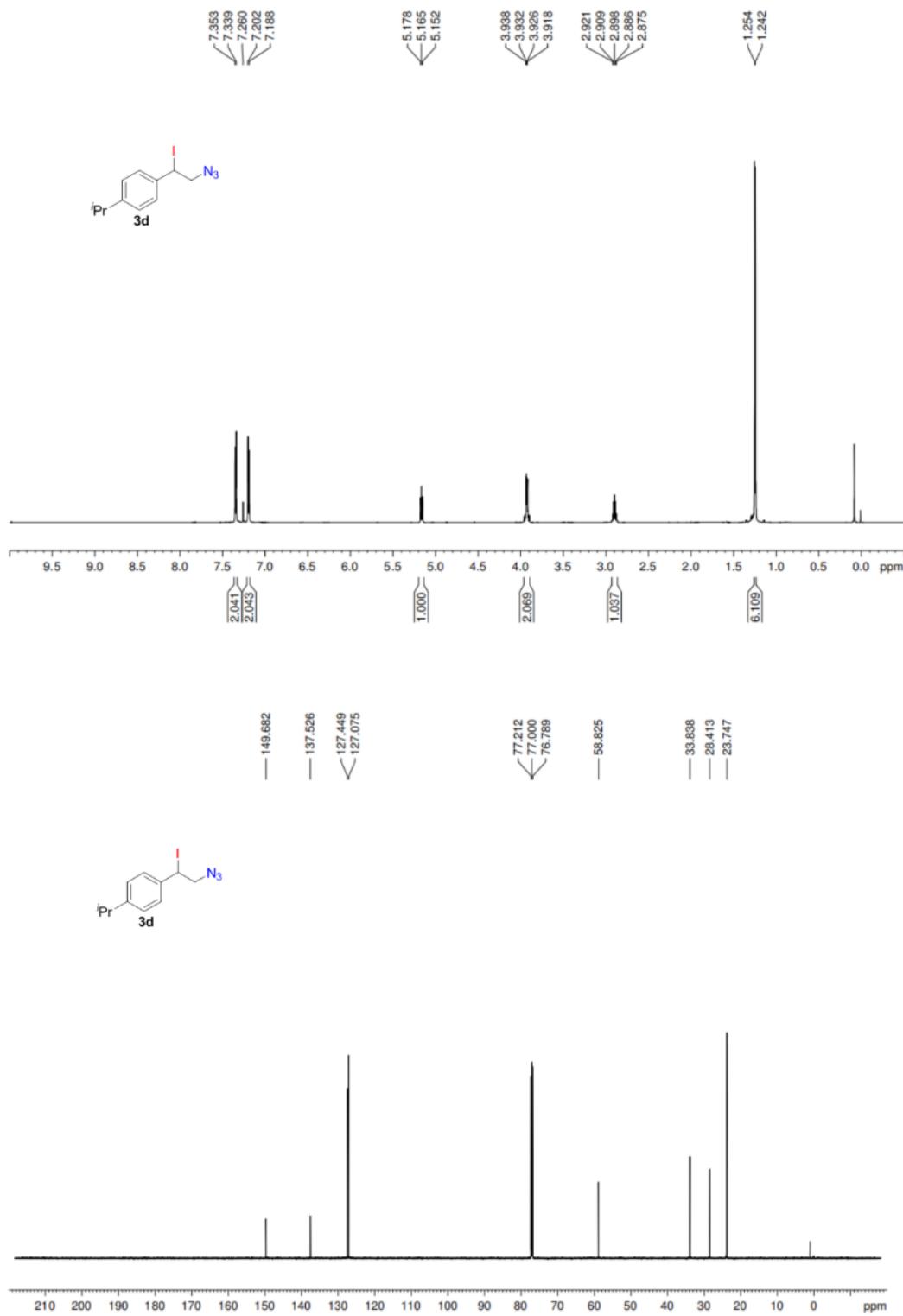


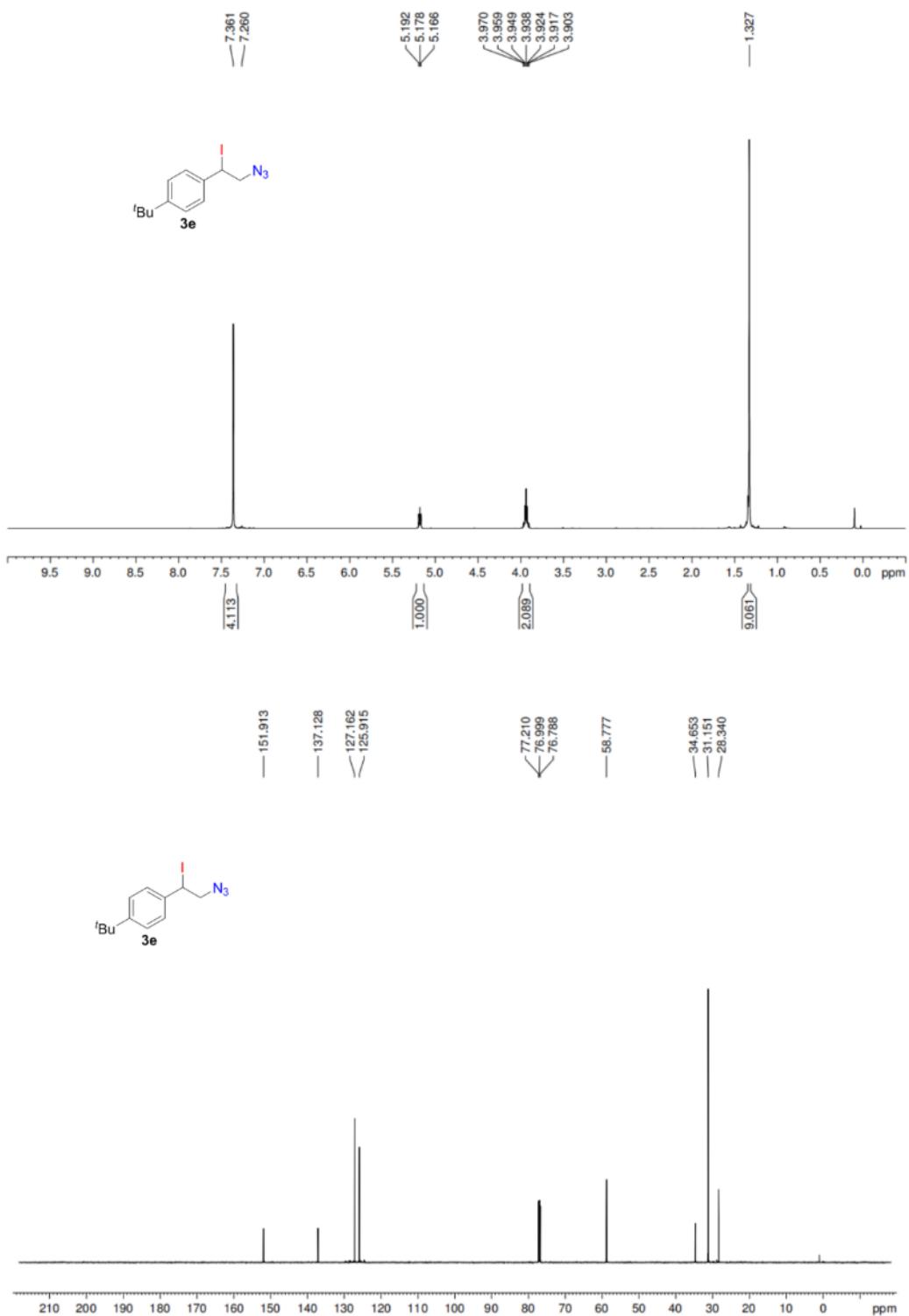


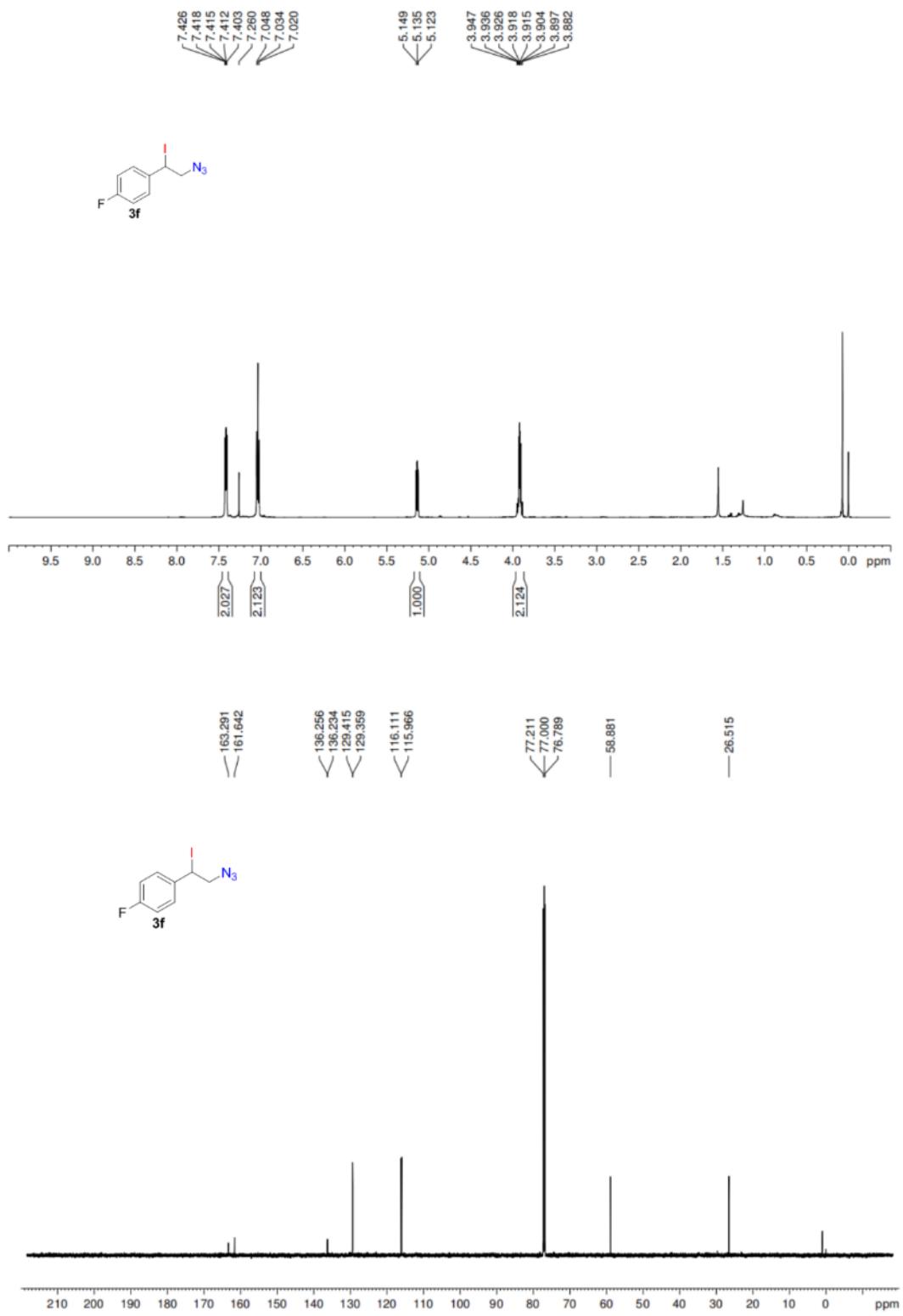


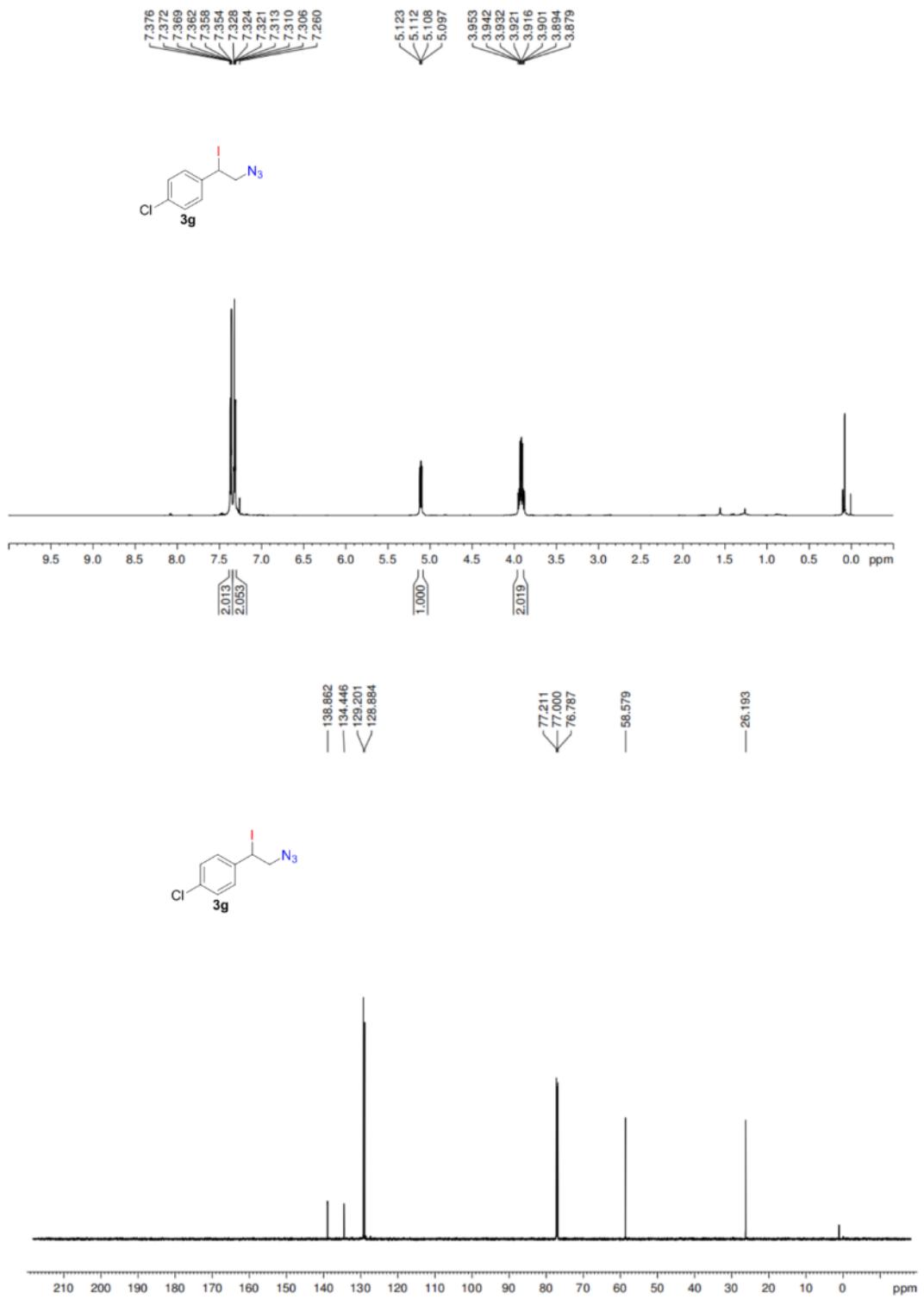


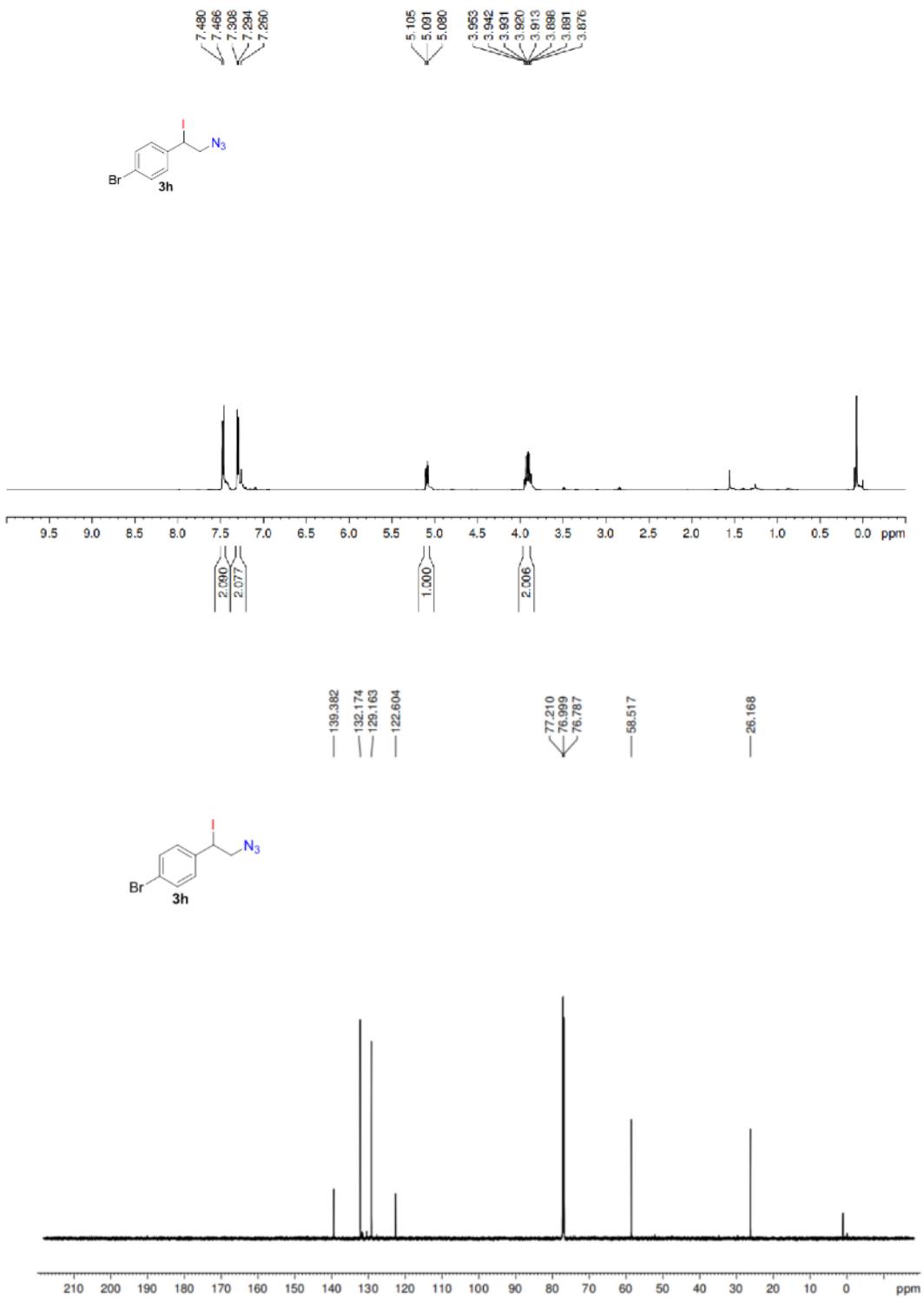


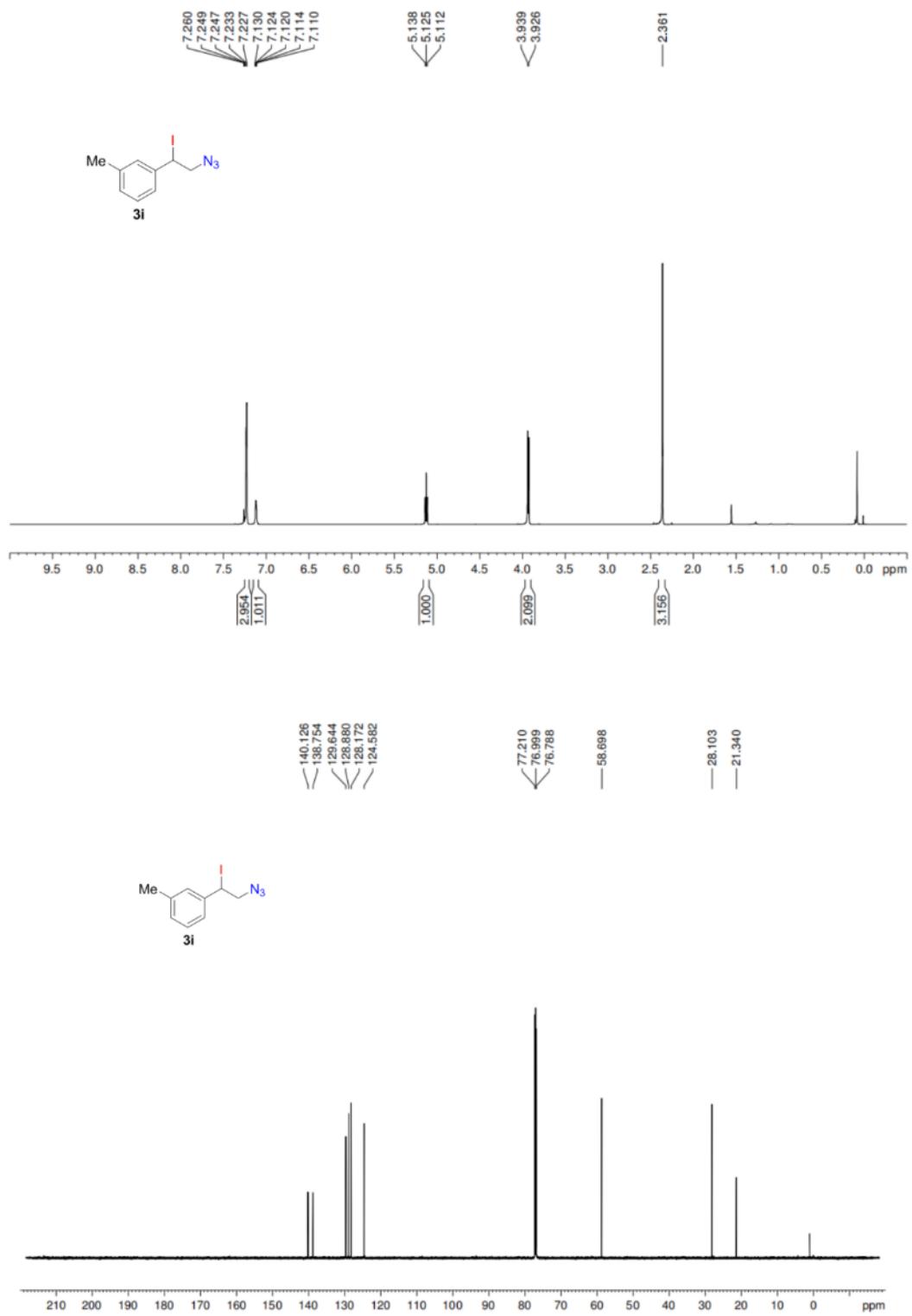


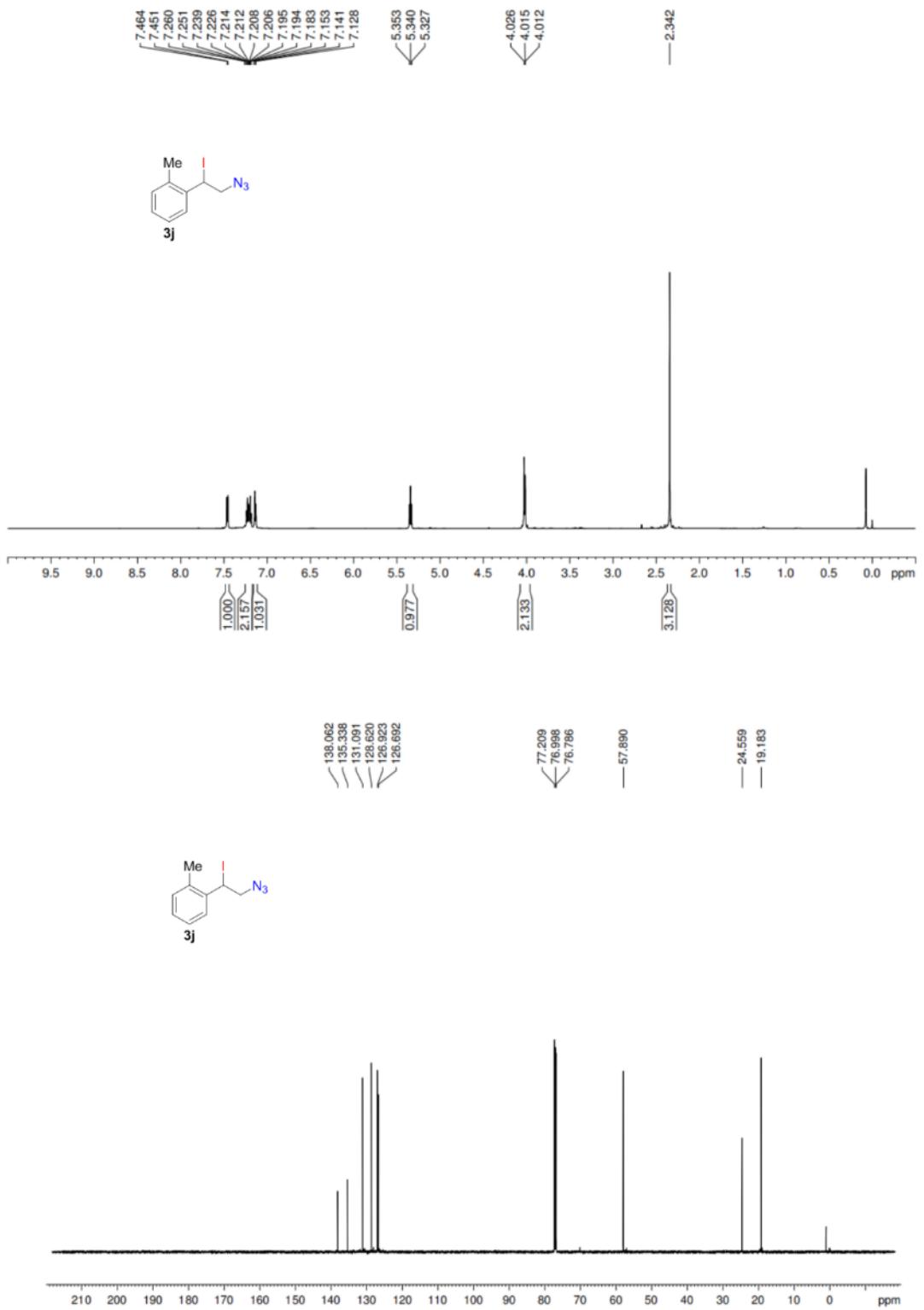


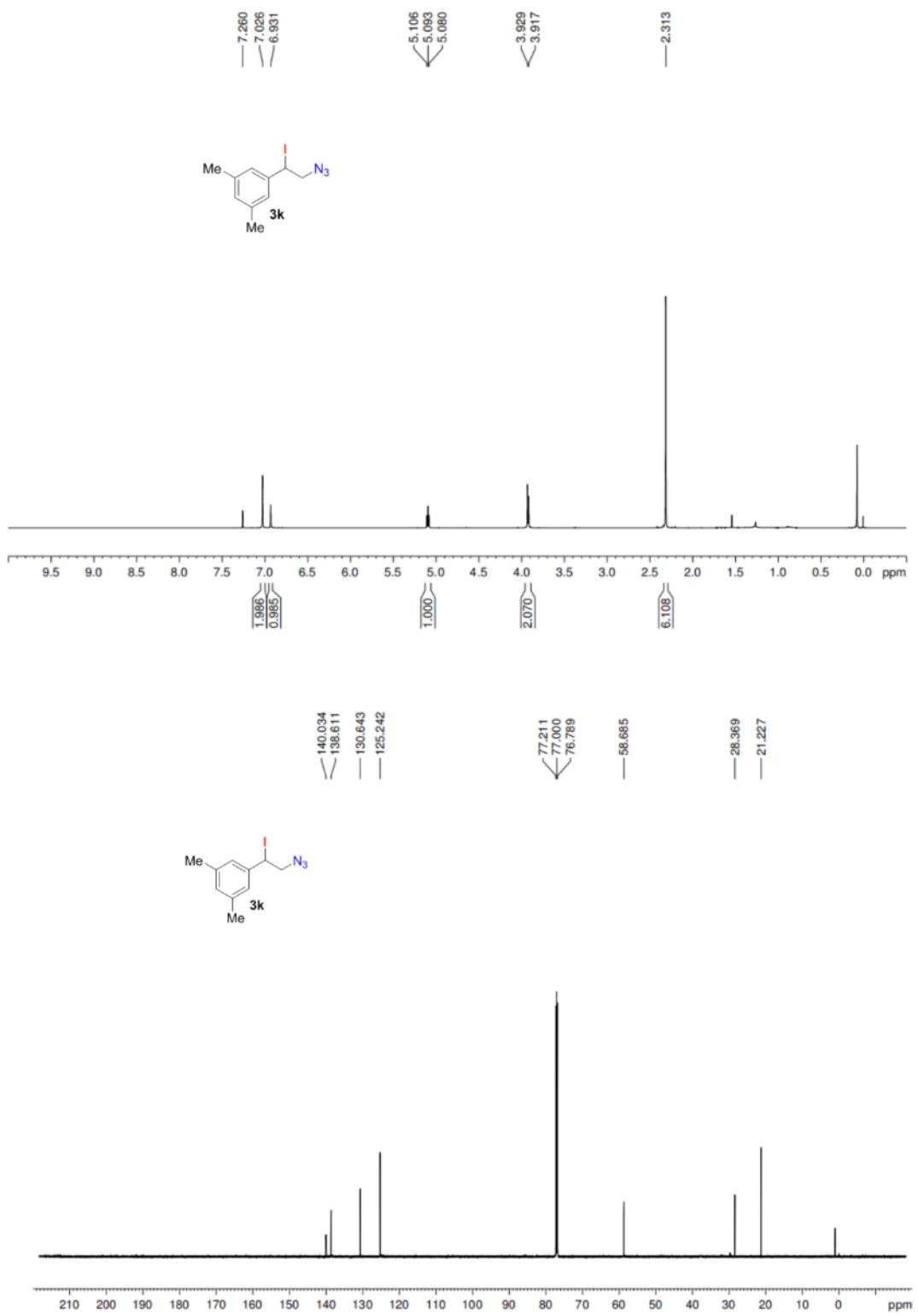


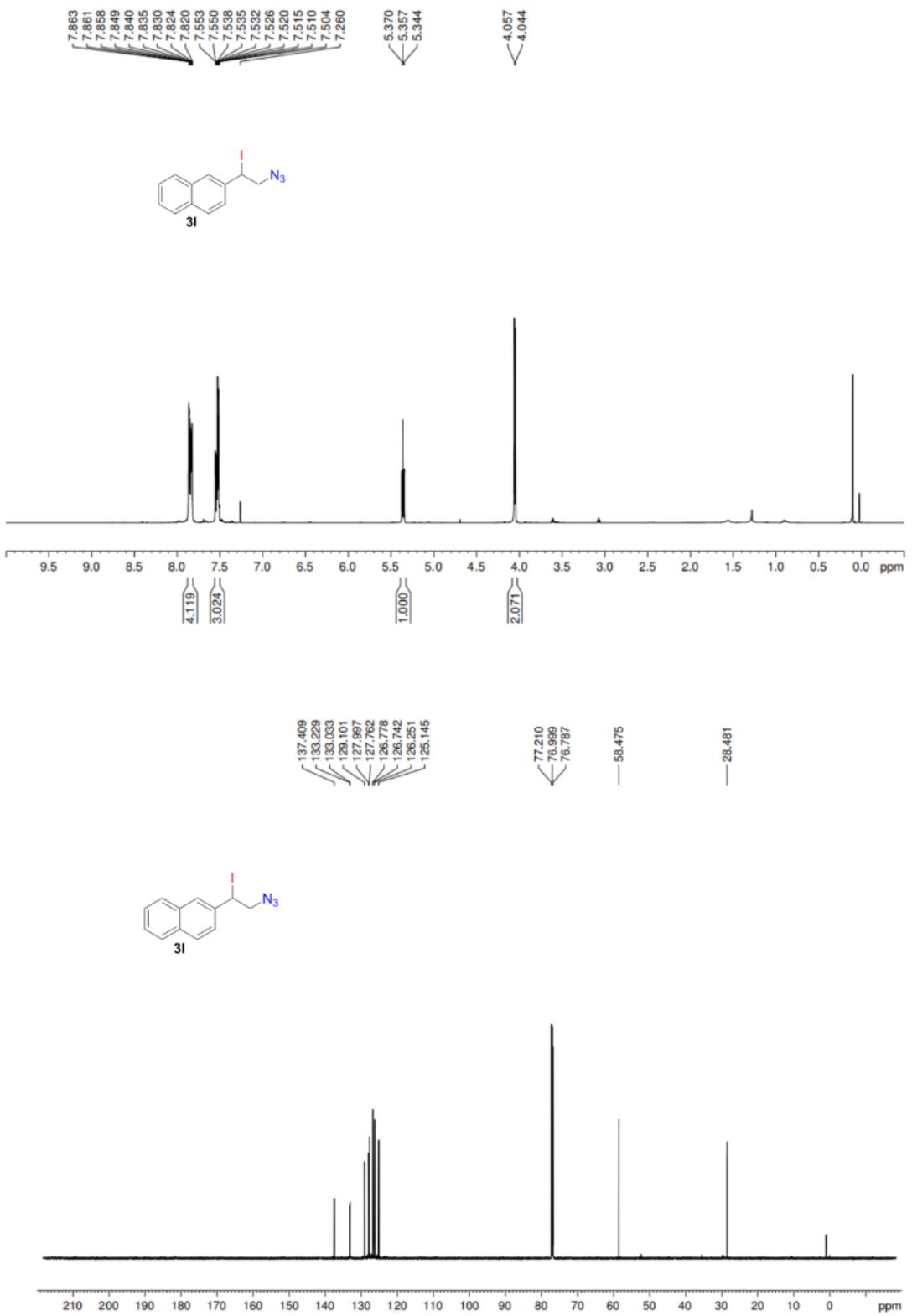


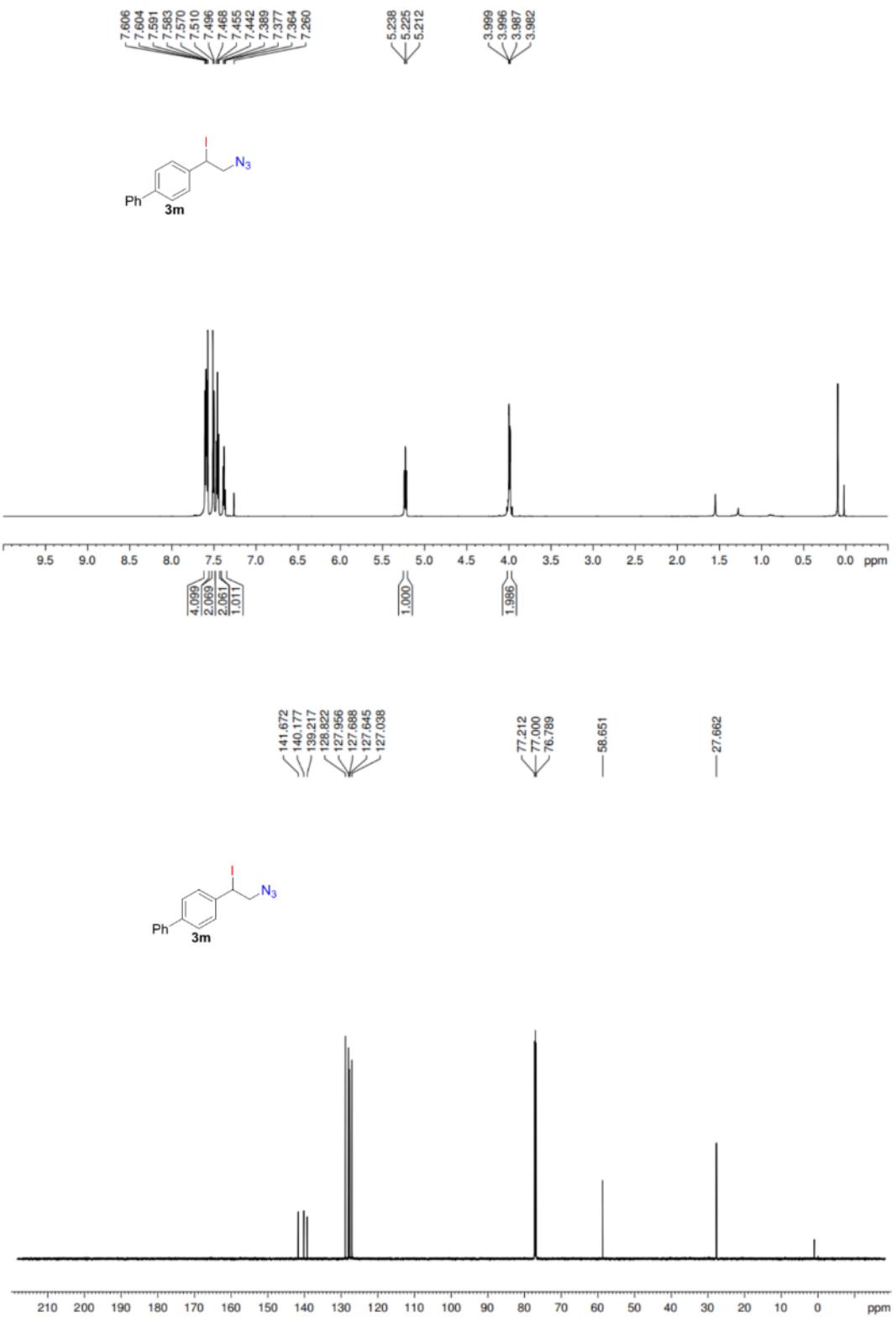


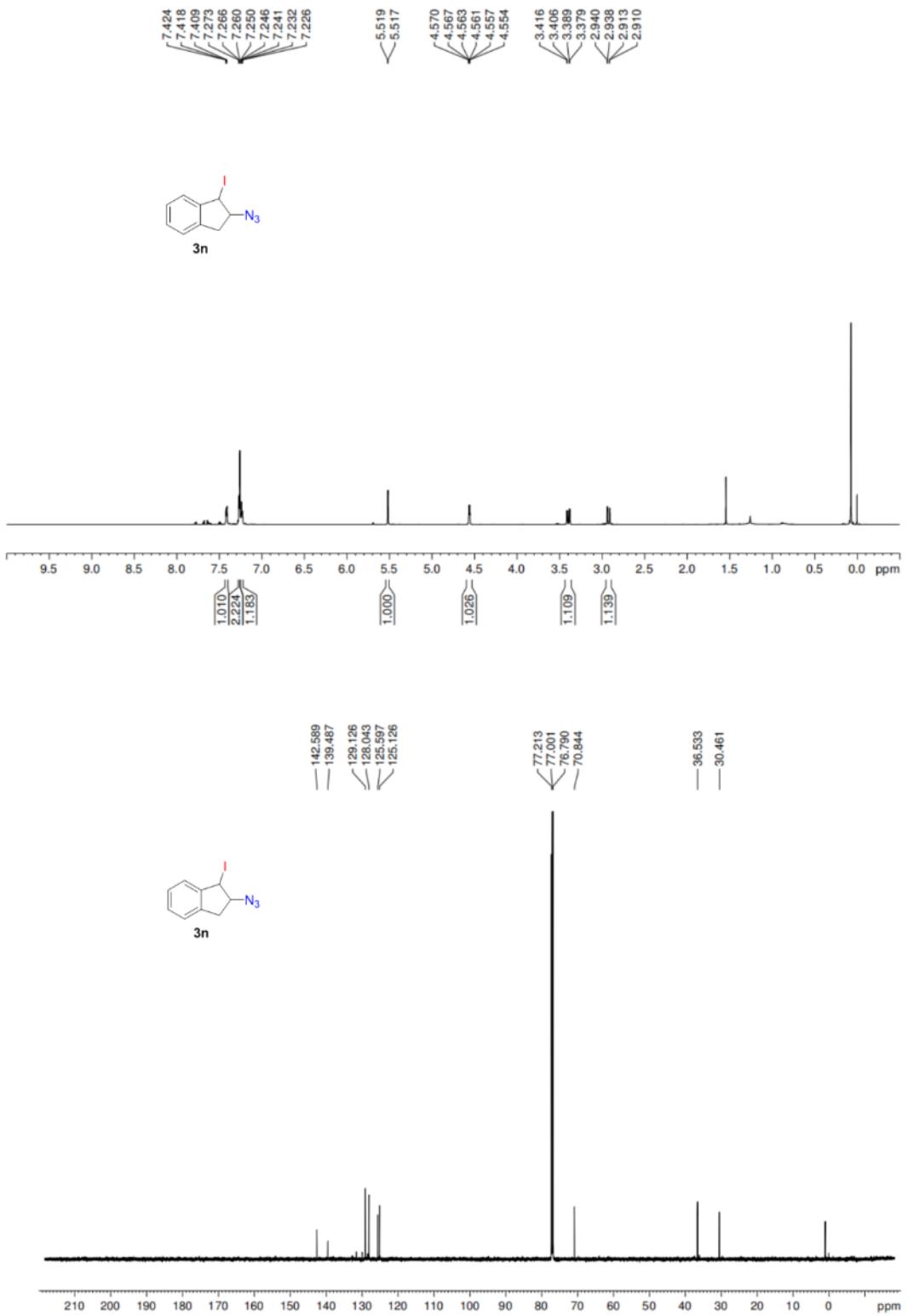


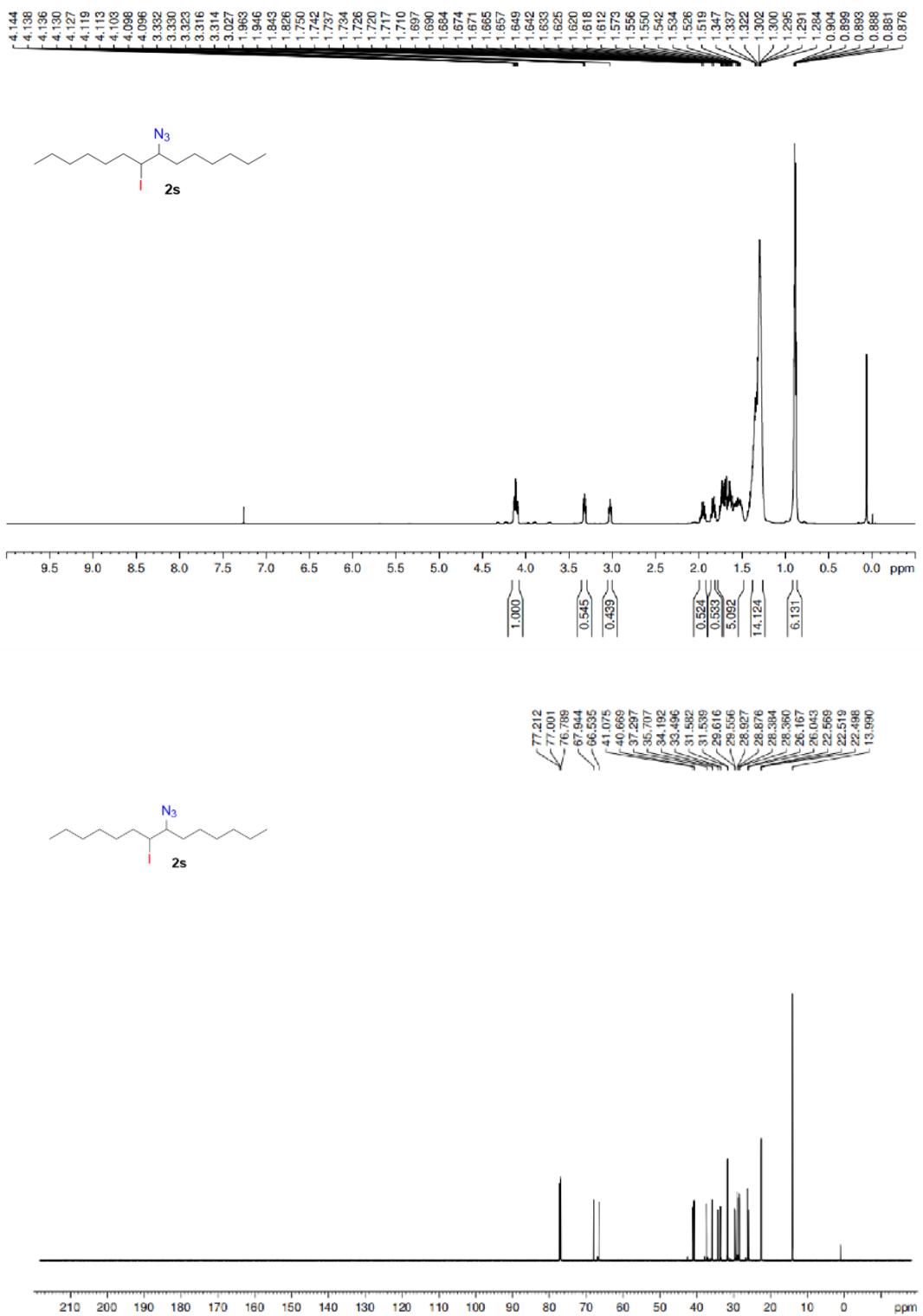


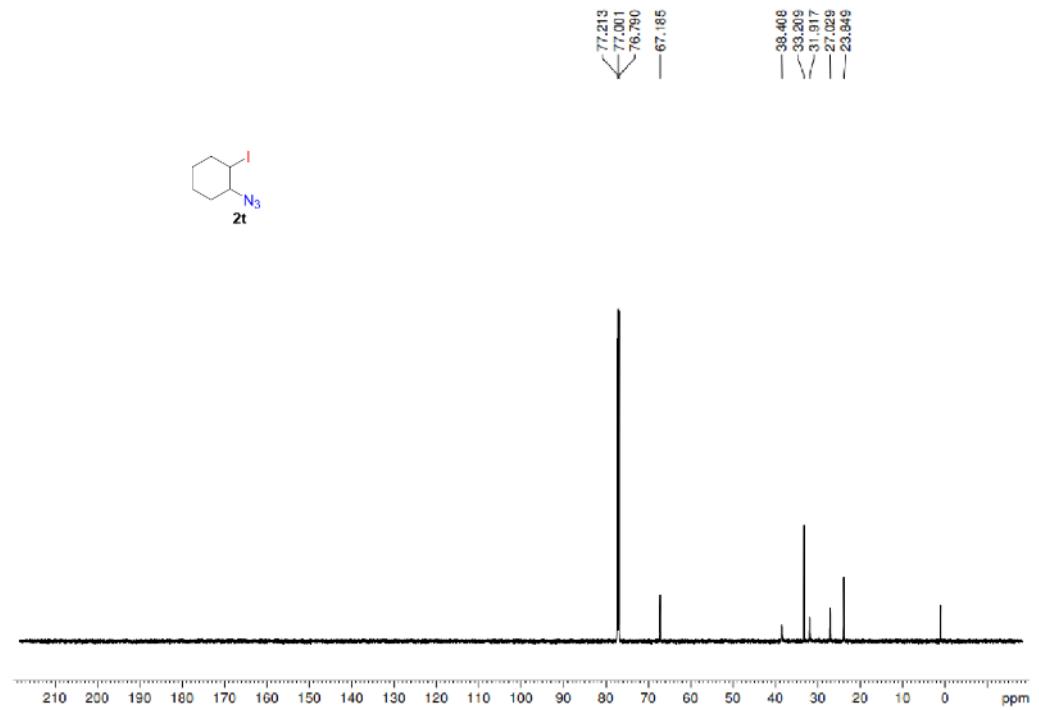
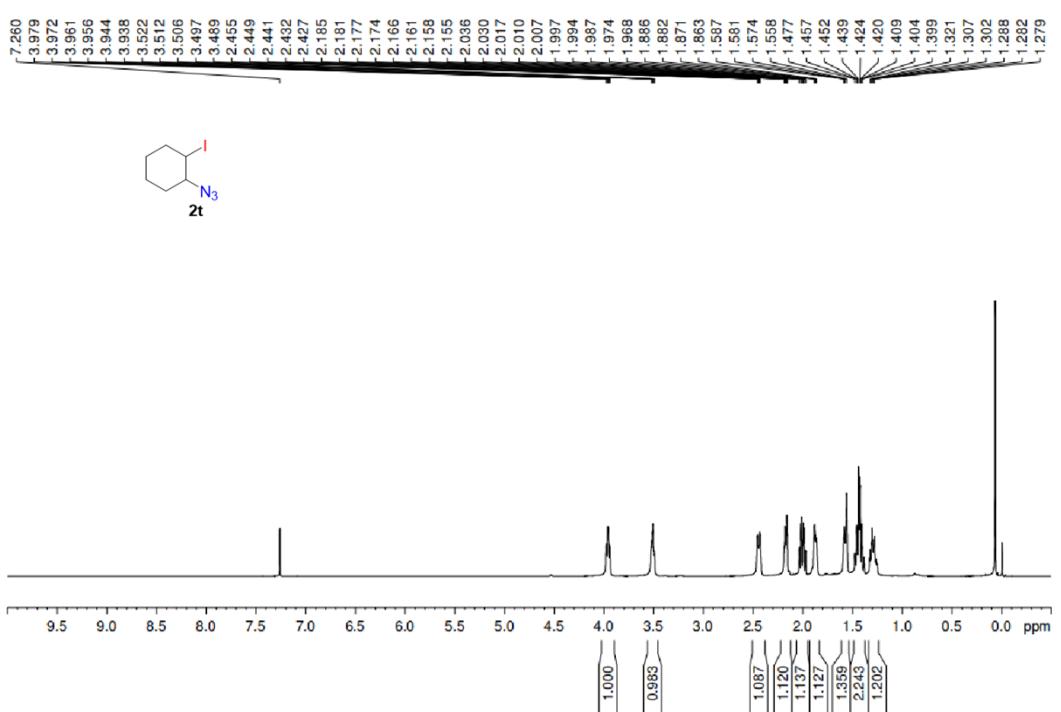


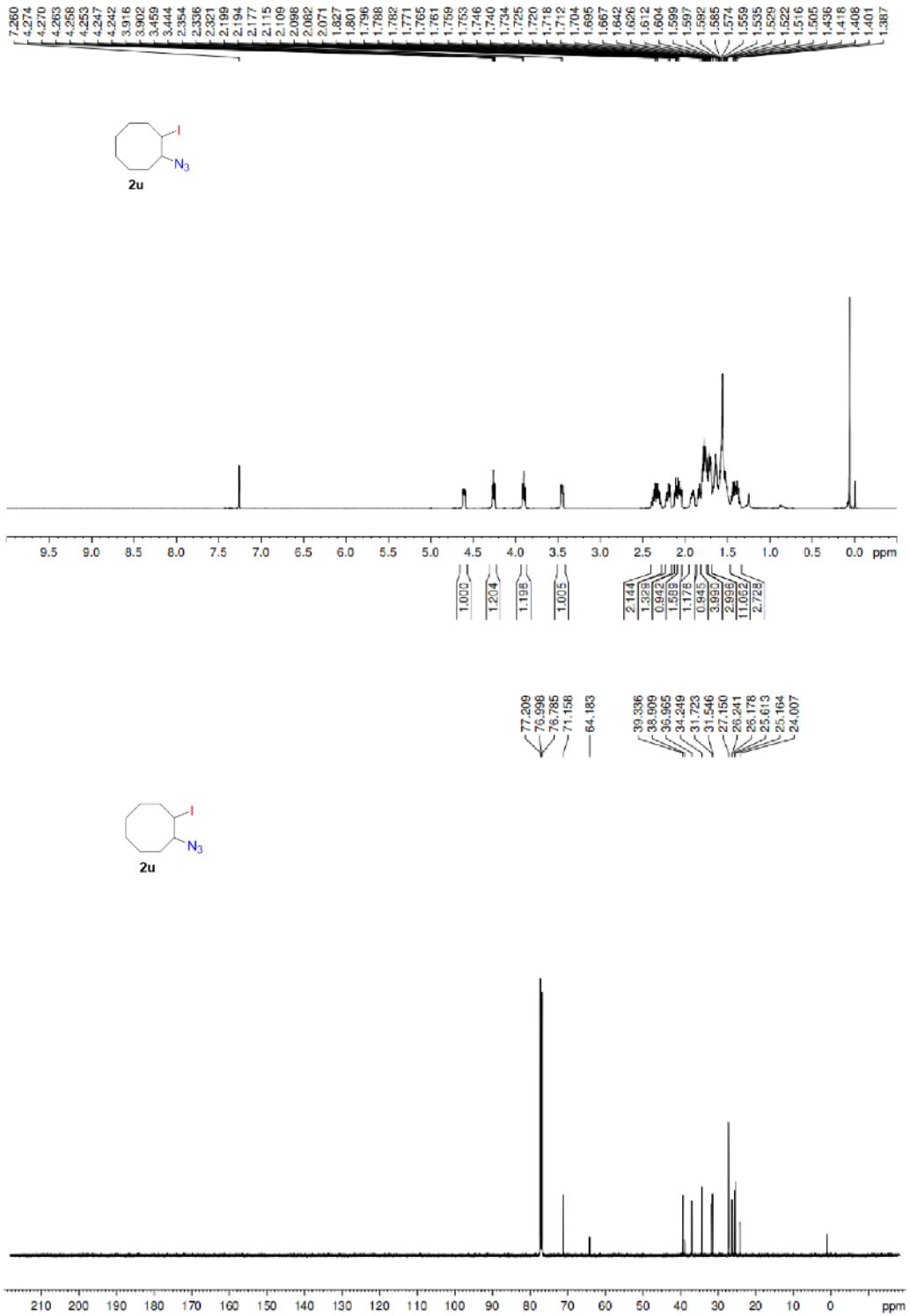


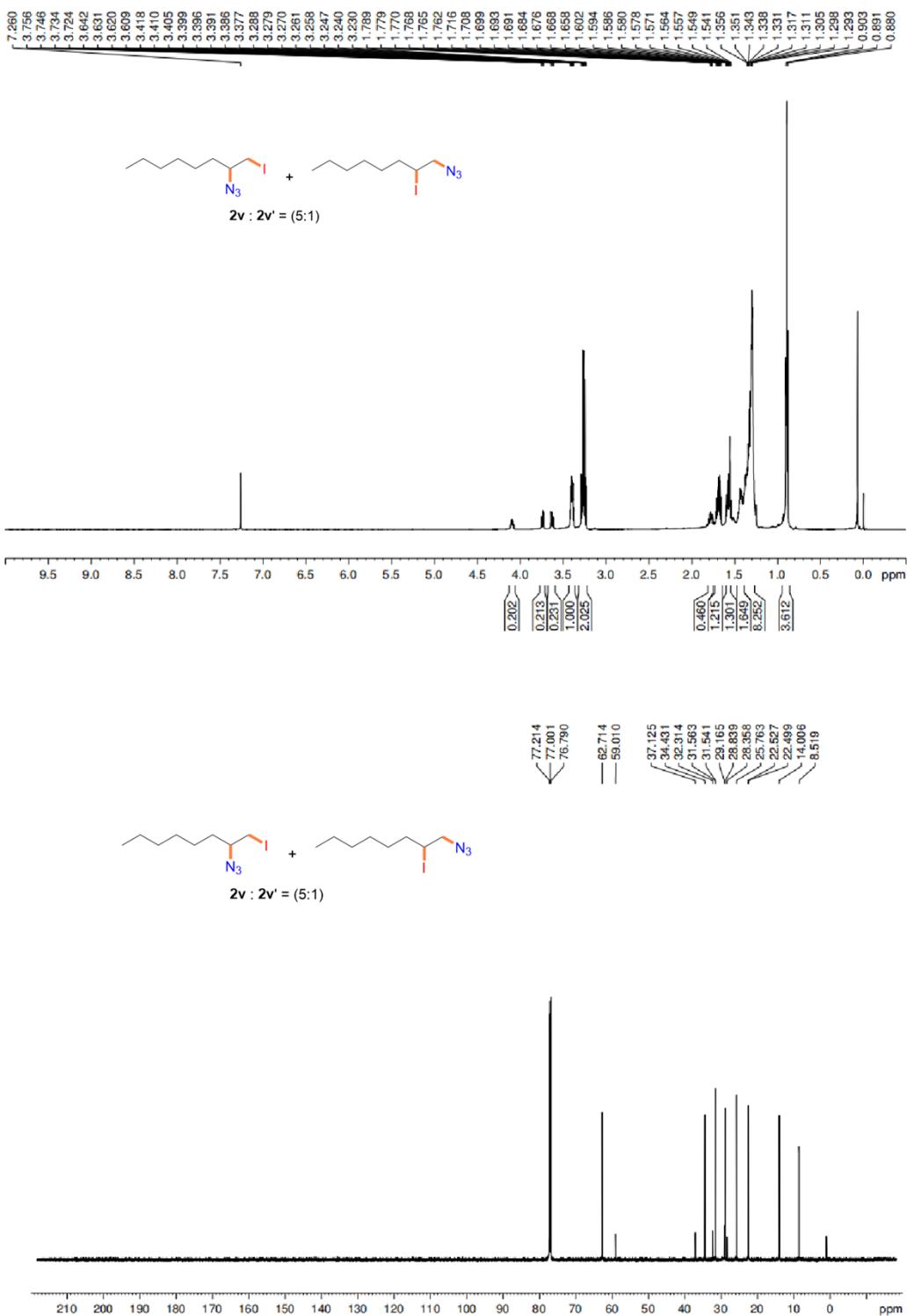


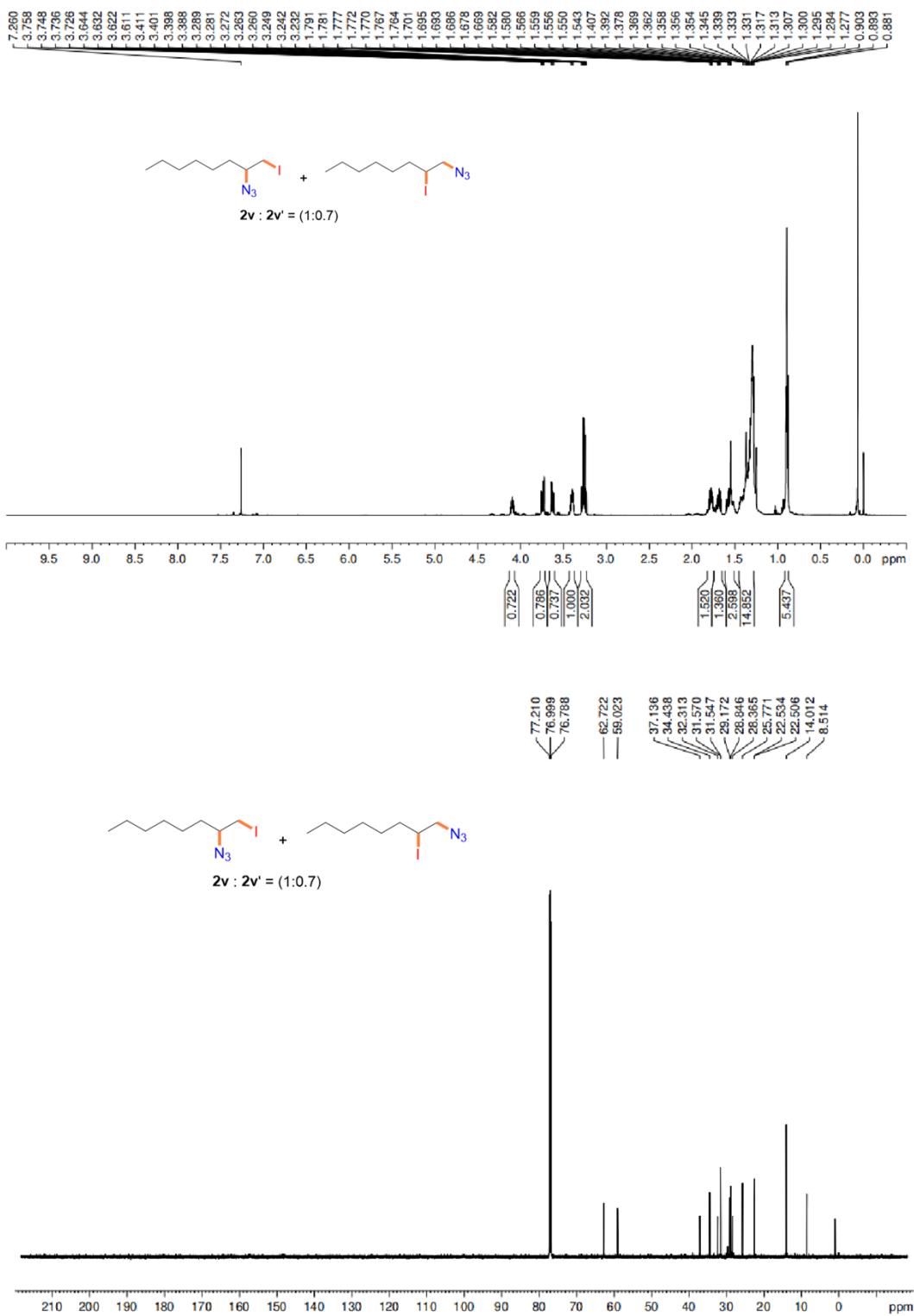


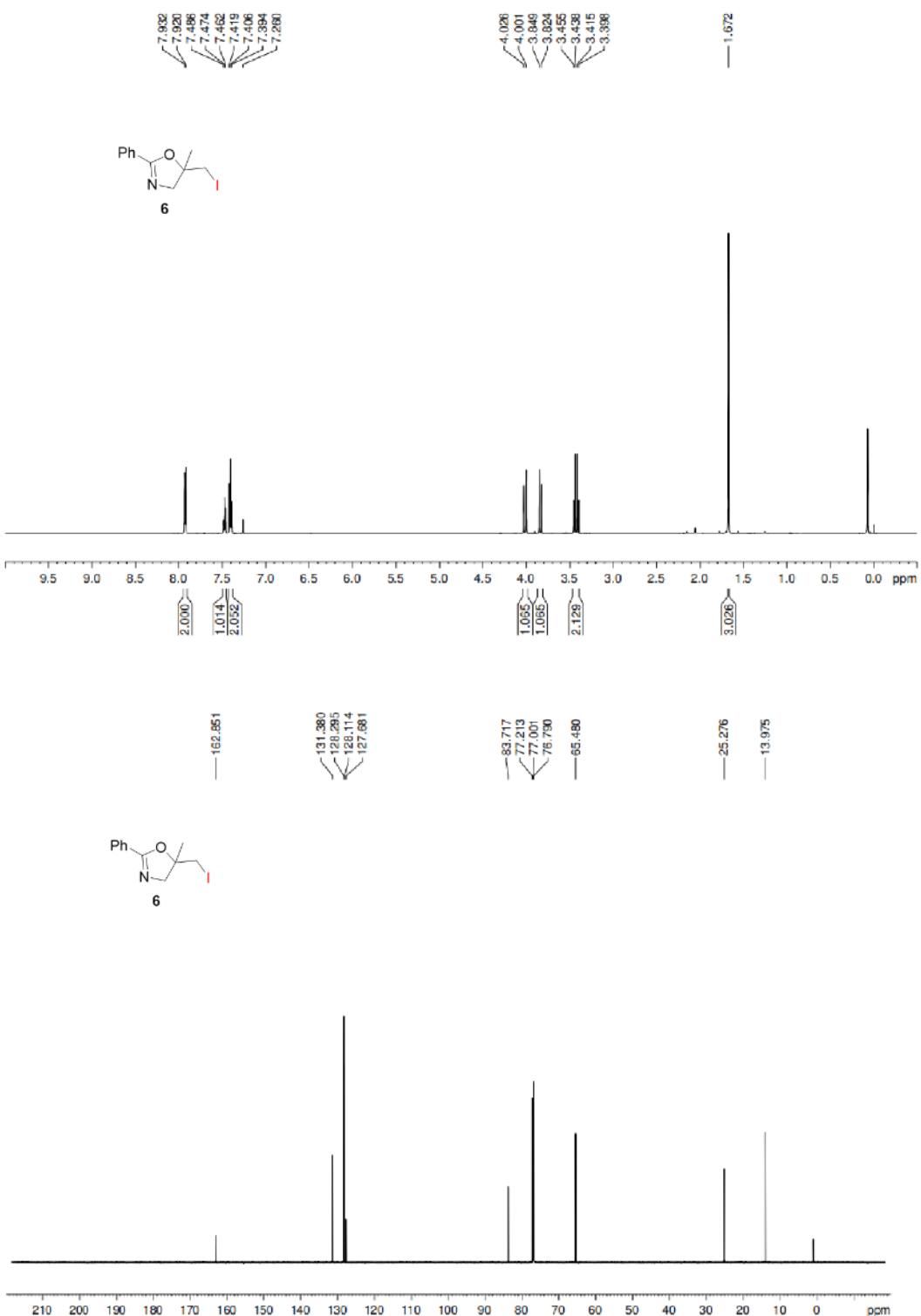


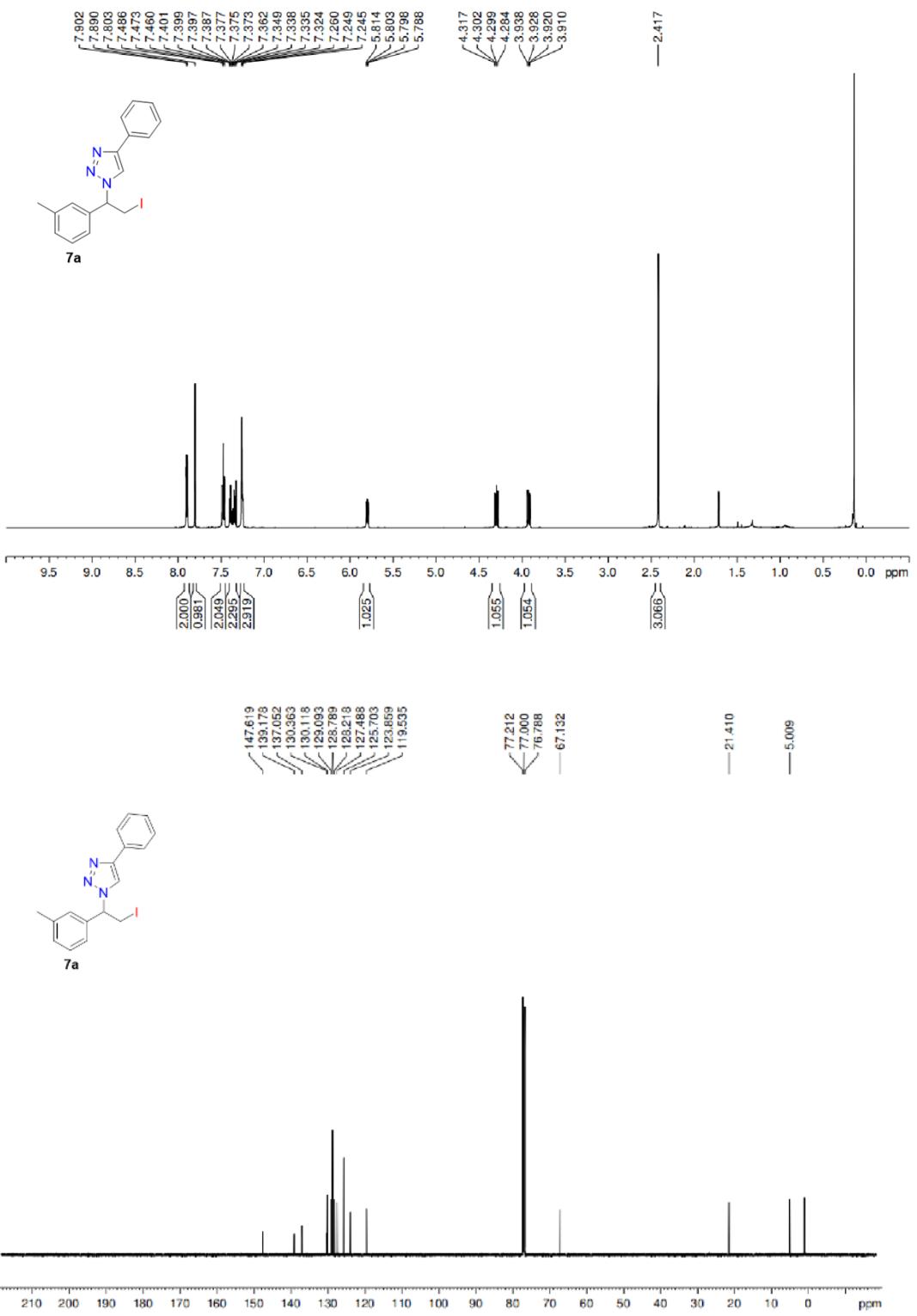


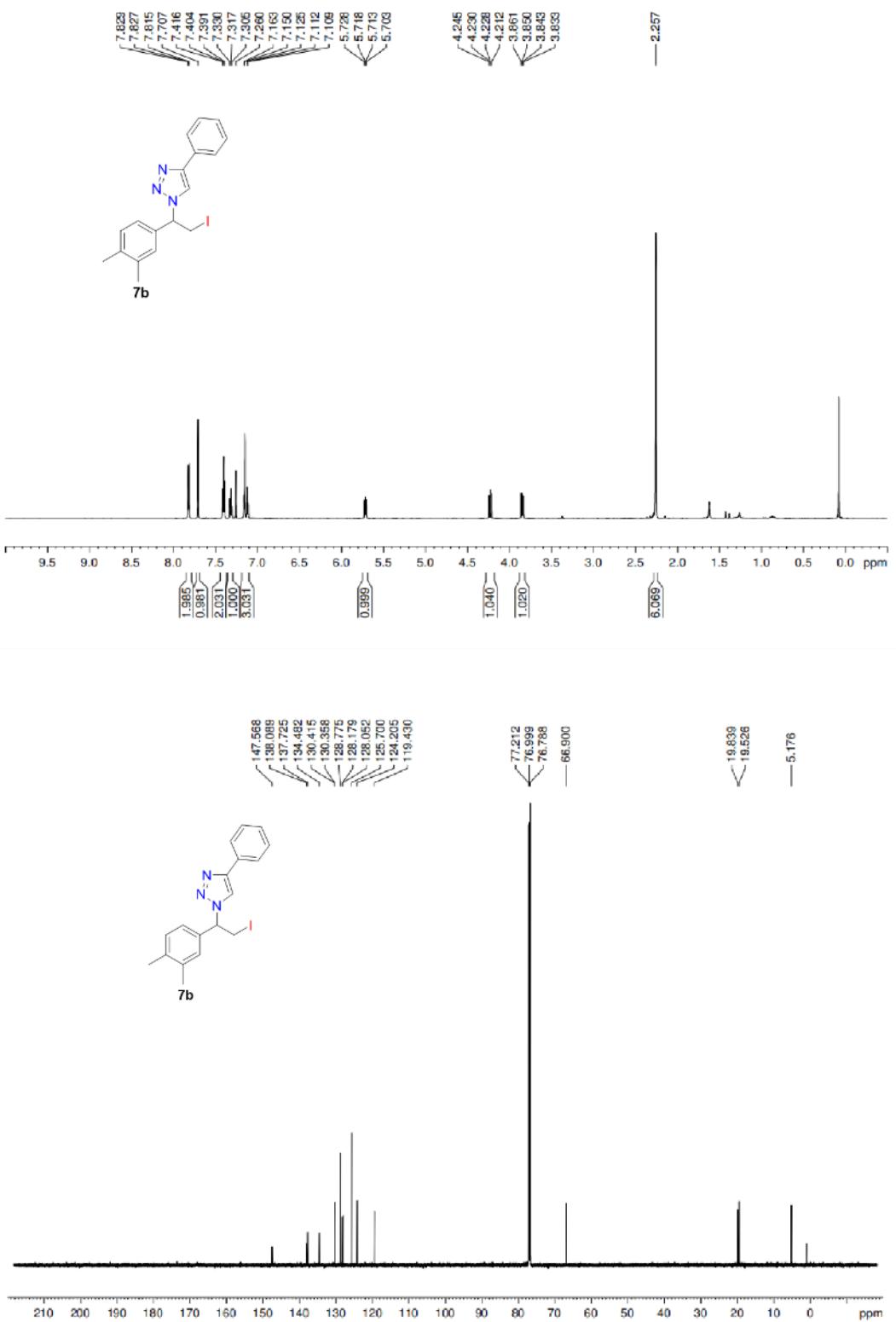


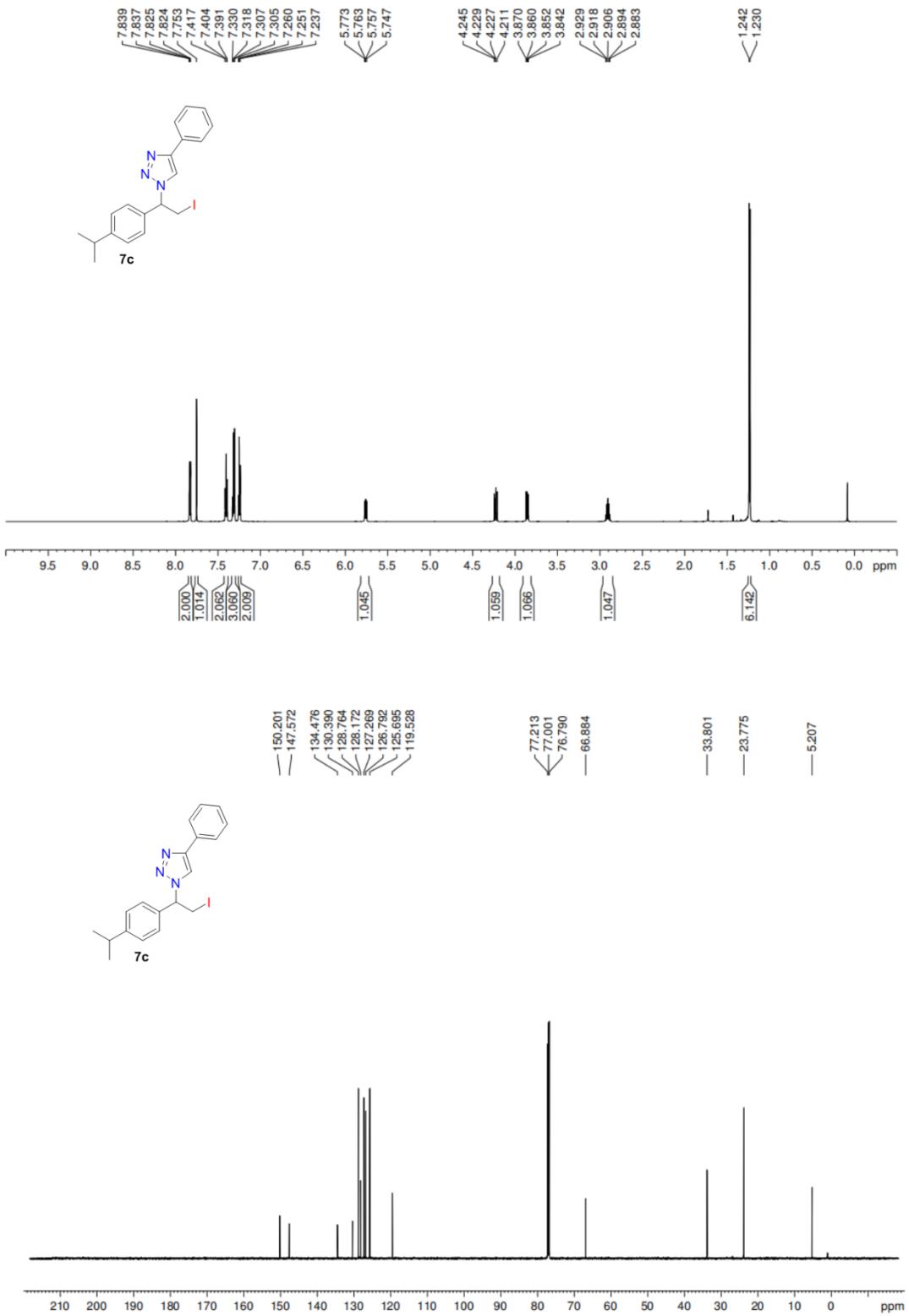


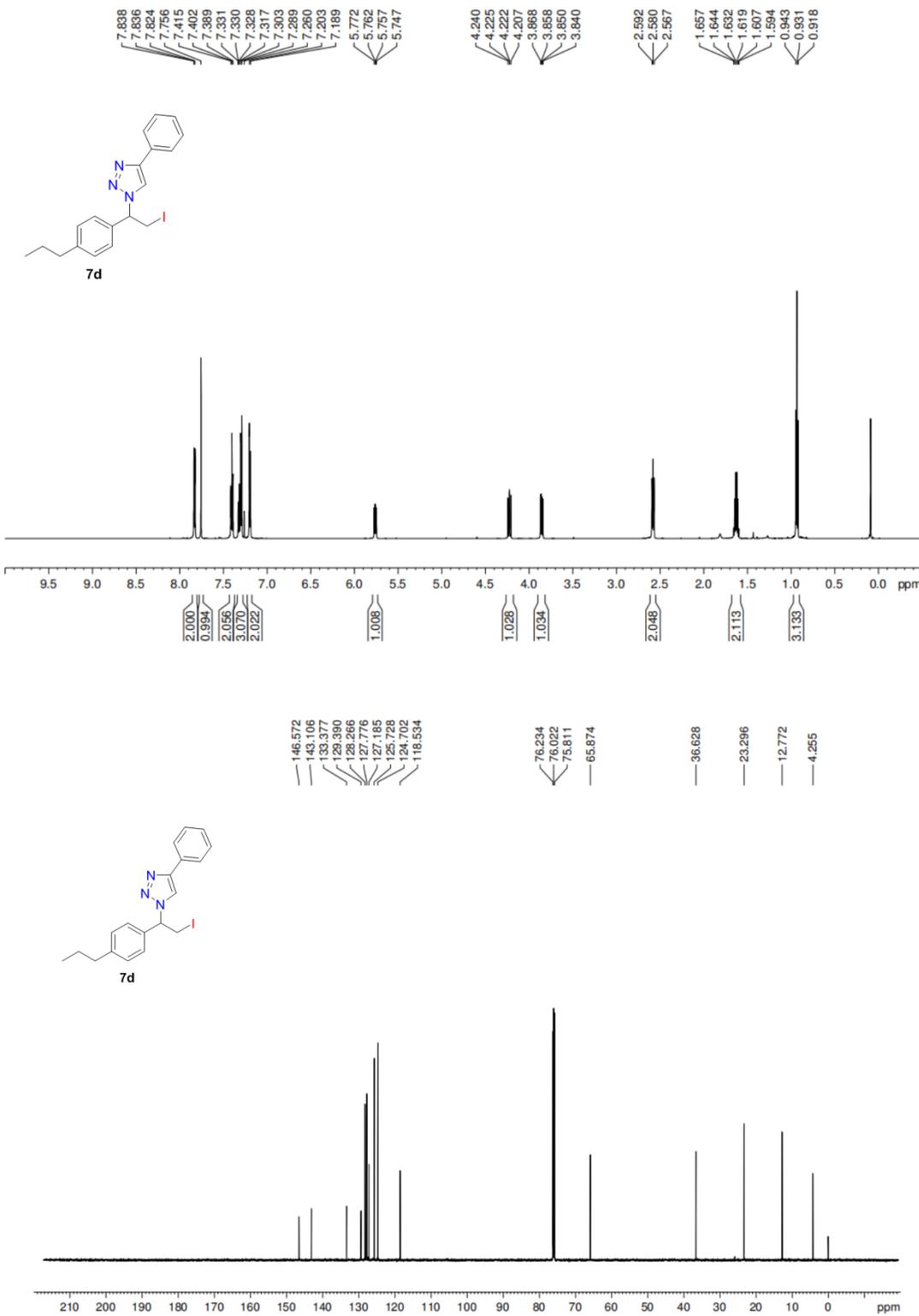


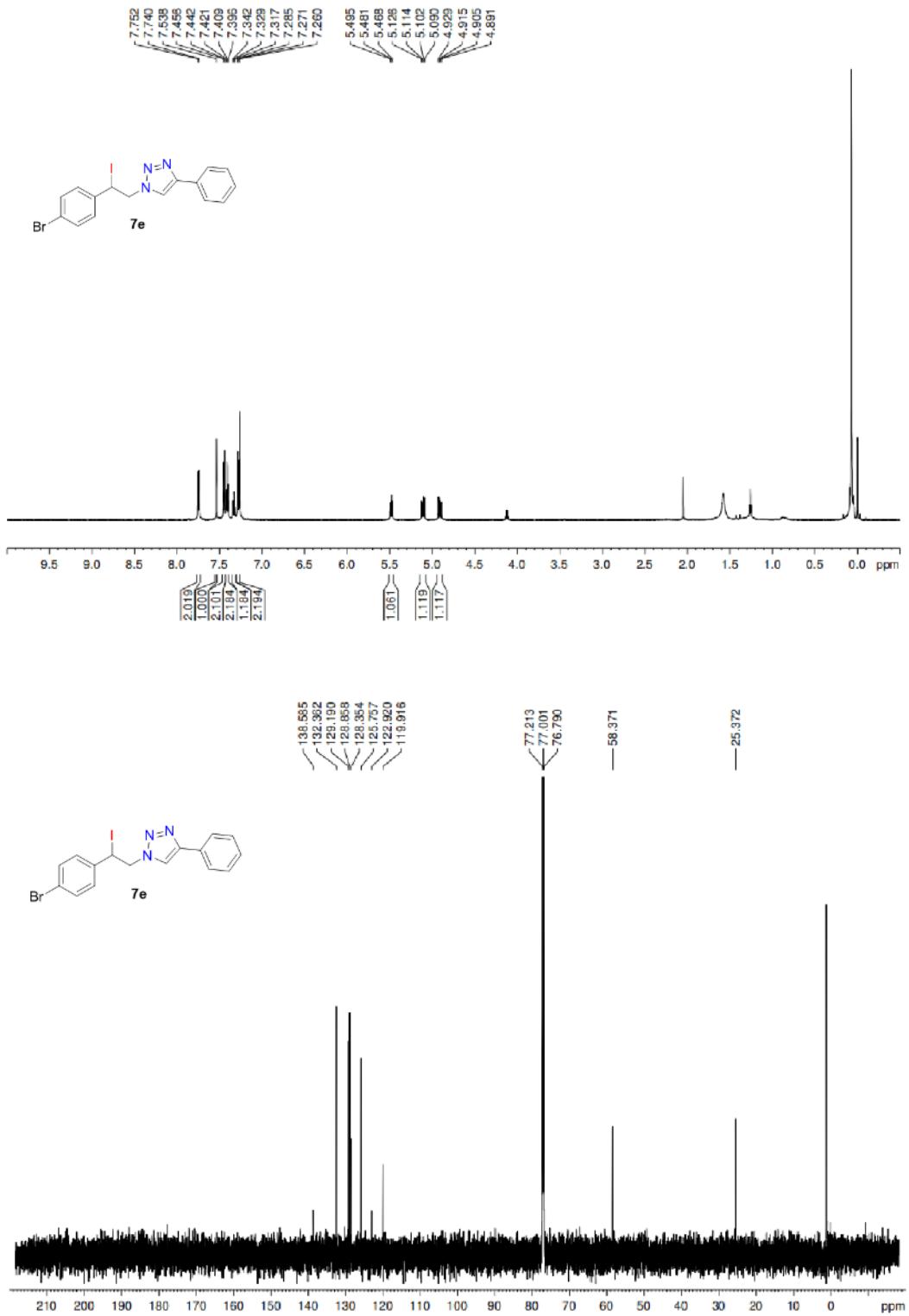


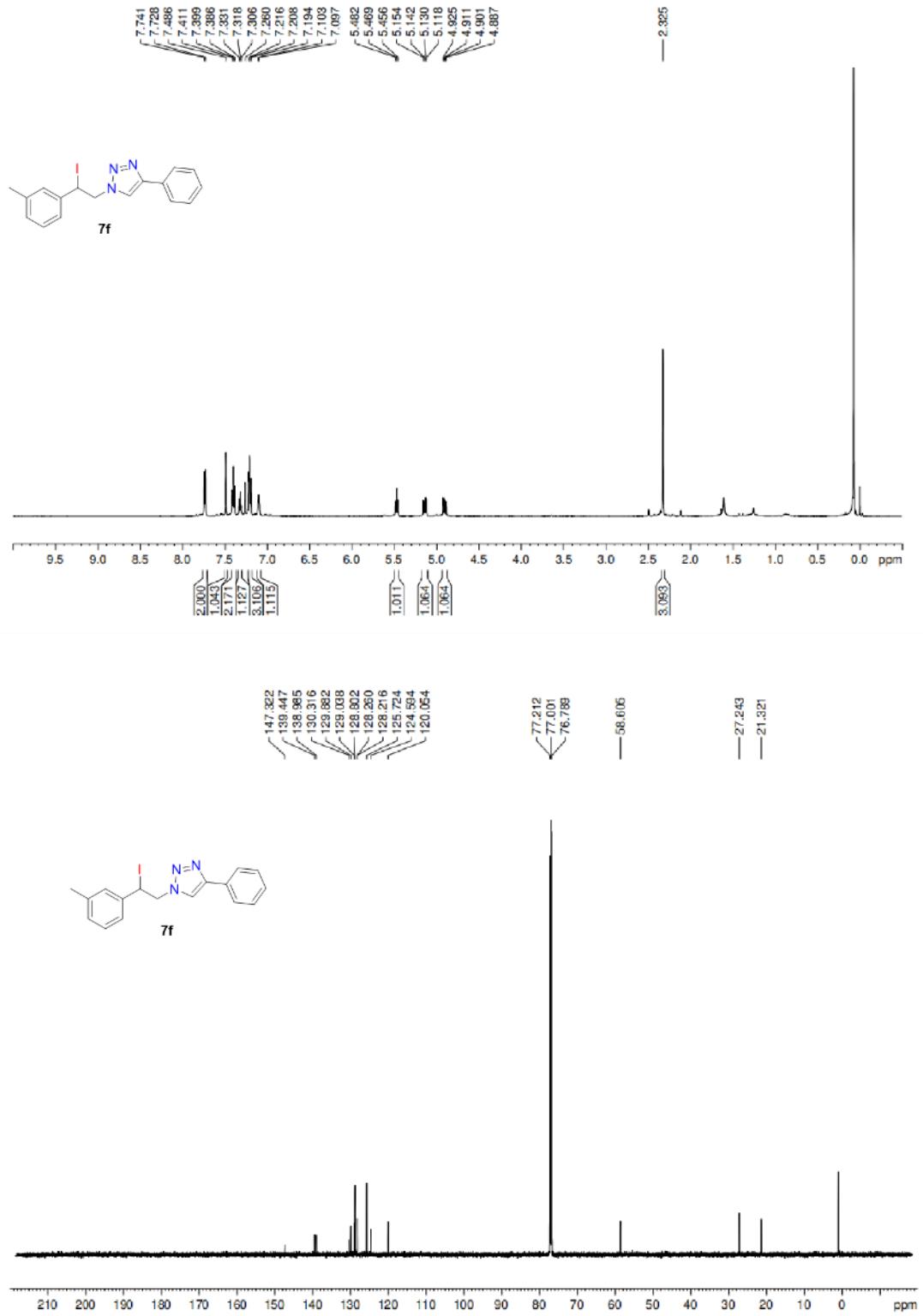


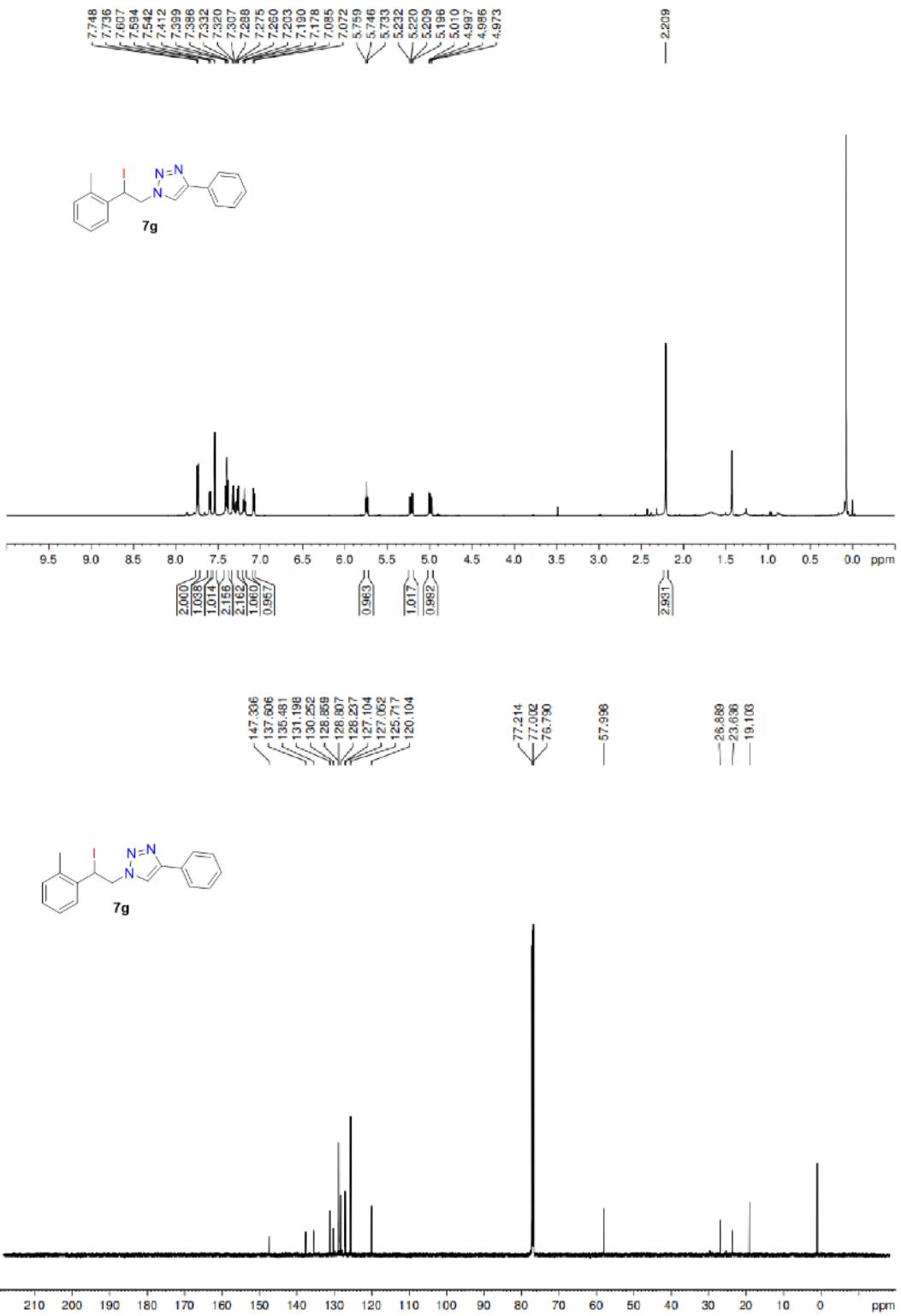


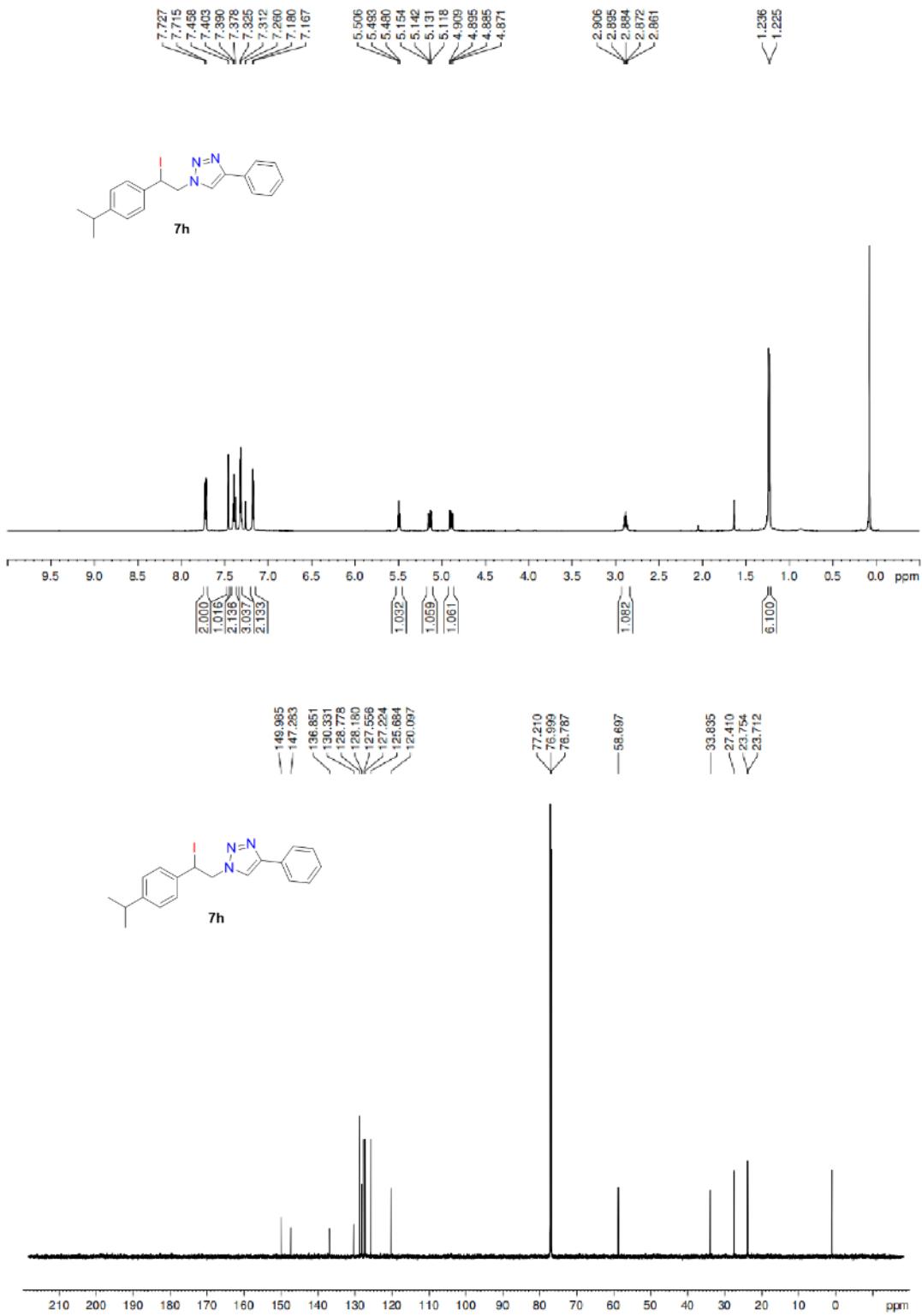


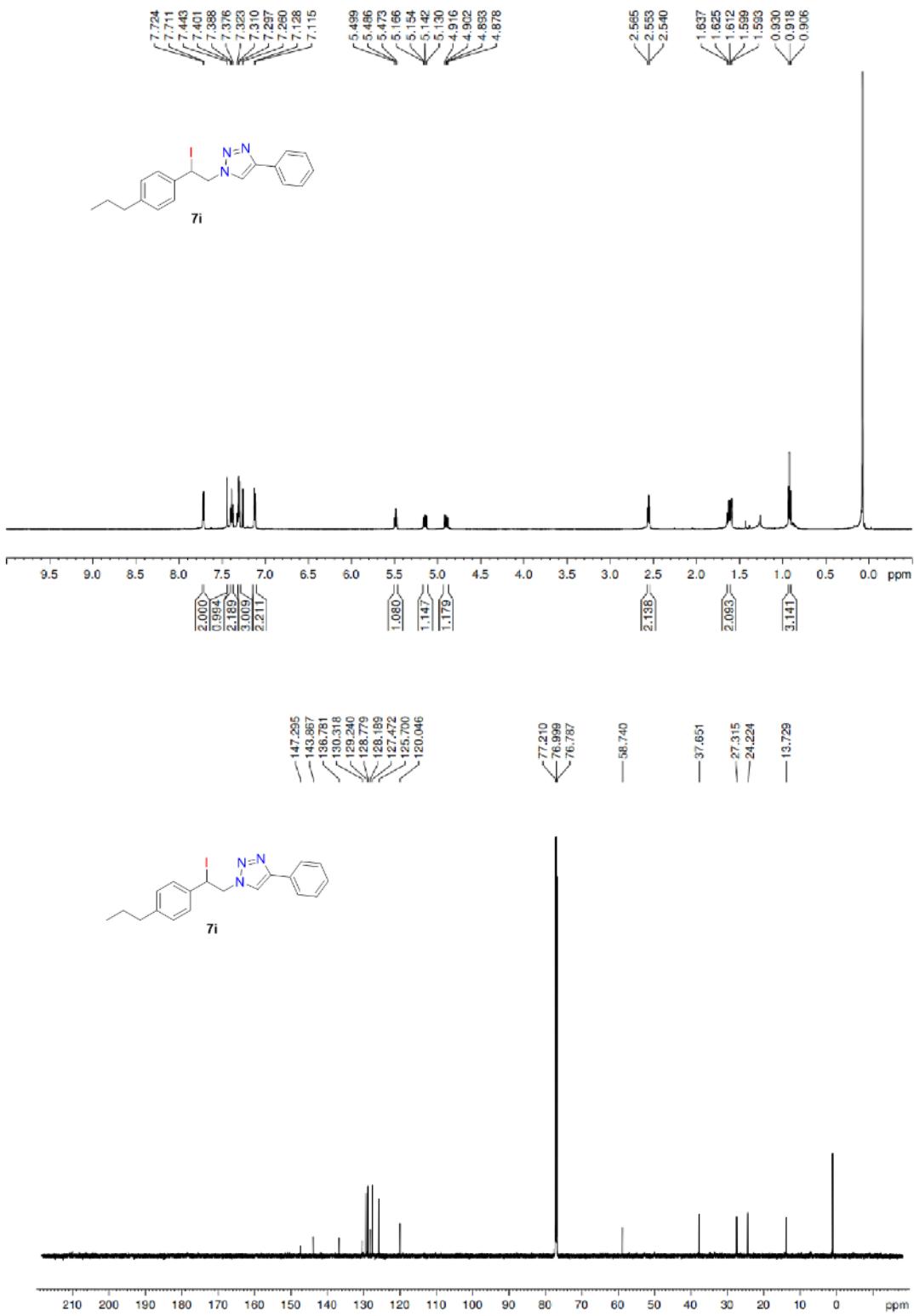


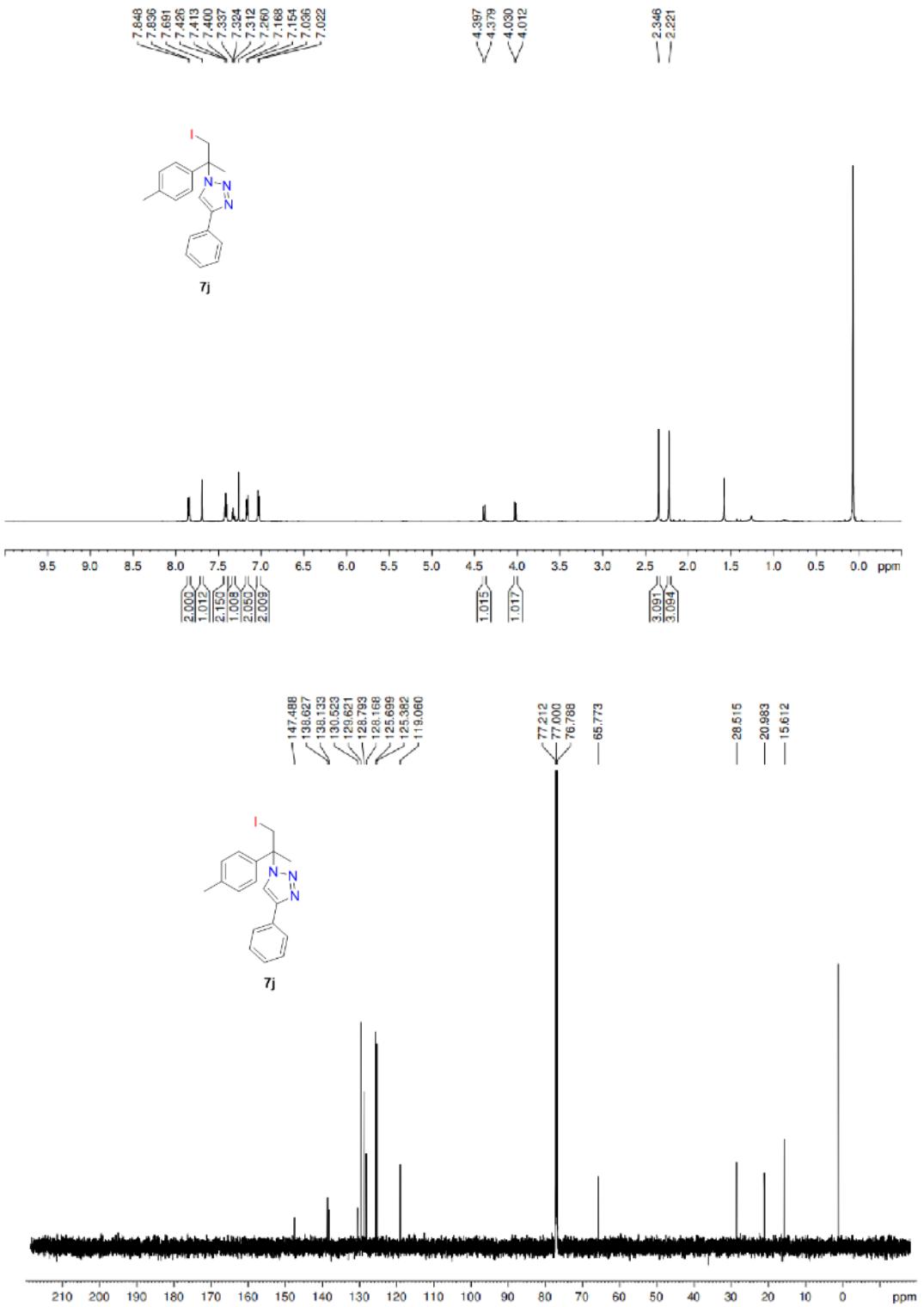


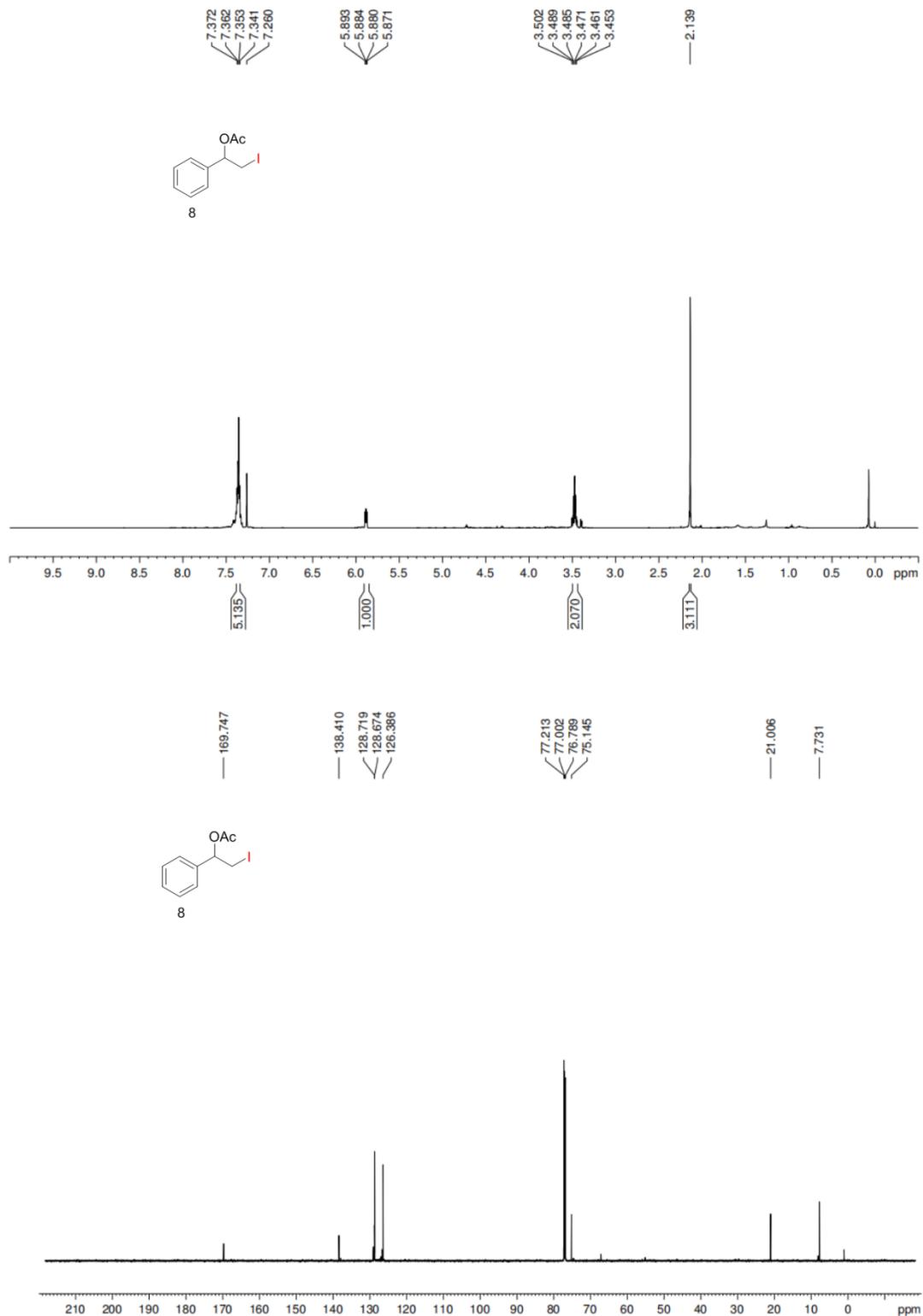












9. References

1. S. Luan, T. Castanheiro and T. Poisson, Electrochemical Synthesis of Iodohydrins, *Adv. Synth. Catal.*, 2022, **364**, 2741–2747.
2. R. S. Dodla, R. R. Thurpu, G. Aakanksha, K. Manoj, and K. Sudhir, Photoswitchable Regiodivergent Azidation of Olefins with Sulfonium Iodate(I) Reagent, *Org. Lett.*, 2019, **21**, 9990–9994.
3. A. K. Tapas, M. Saikat and M. Prasenjit, PIDA-I₂ Mediated Direct Vicinal Difunctionalization of Olefins: Iodoazidation, Iodoetherification and Iodoacyloxylation, *Org. Biomol. Chem.*, 2016, **14**, 4654–4663.
4. C. V. Pandurang, K. U. Pratibha, S. Gurunath, S. Arumugam, Regiospecific Azidoiodination of Alkenes with Sodium Periodate, Potassium Iodide, and Sodium Azide: A High-Yield Synthesis of β-Iodoazides, *Synthesis.*, 2010, **22**, 3879–3882.
5. C. Massimo, E. Francesco, M. C. Maria and R. Ornelio, Simple and Regioselective Azidoiodination of Alkenes using Oxone®, *Tetrahedron Letters.*, 2002, **43**, 1201–1203.
6. T. O. Alexander, K. B. Igor, K. A. Vladimir and N. I. Gennady, Facile Method for the Synthesis of Vicinal Azidoiodides by the Reaction of the NaN₃-I₂ System with Unsaturated Compounds, *Synthetic Communications.*, 2008, **38**, 3797–3809.
7. H. Tatsuki, Y. Shohei, H. Shoji, Iodoazidation of Alkenes by Using Iodine Pentafluoride-Pyridine-Hydrogen Fluoride and Trimethylsilyl Azide, *Synthesis* 2016., **48**, 1353–1358.
8. G.-Q Liu, C.-H Yang, Y.-M Li, Modular Preparation of 5-Halomethyl-2-oxazolines via PhI(OAc)₂-Promoted Intramolecular Halooxygenation of N-Allylcarboxamides, *J. Org. Chem.*, 2015, **80**, 11339–11350.
9. T. N. Muhammad, Q. Riaz-ul, A. Ahmad, C. Eunae, J. Seunho, Continuous Process for Click Reactions using Glass Micro-Reactor Functionalized with β-Cyclodextrin, *Tetrahedron Letters.*, 2013, **54**, 3268–3273.