Supporting Information

Electrochemical isothiocyanation of primary amines

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Material and methods

General Experimental Procedures

All reactions were carried out under aerobic conditions unless otherwise stated. All solvents and commercially available reagents were purchased from standard vendors and used without further purification unless otherwise stated. Electrolyses were performed using an IKA Electrasyn 2.0 using carbon graphite working electrode and nickel counter electrode. Analytical thin-layer chromatography (TLC) was performed using silica gel plates (0.25 mm thickness) on an aluminium support. Visualization was accomplished by irradiation with a UV lamp and/or staining with either $KMnO_4$ or ninhydrin. Column chromatography was performed over Silica gel 60 Å (40-63µ mesh) using a CombiFlash Rf Lumen automatic flash chromatography system. Residual solvent was removed using a static oil pump (< 10 mbar). The cooling of reaction mixtures was achieved using an ice bath (0 °C).

NMR spectra were obtained using a JEOL ECZR 400 (^{1}H 399.78 MHz; ^{19}F 376.17 MHz; ^{13}C 100.53 MHz) or ECA 500 (^{1}H 500.16 MHz; ^{13}C 125.77 MHz) spectrometer and are reported relative to the residual solvent resonances. All heteronuclear NMR spectra were ^{1}H-decoupled and recorded at room temperature unless otherwise stated. Data for ^{1}H NMR spectra are reported as follows: chemical shift (δ, ppm), coupling constant (Hz), multiplicity (s, singlet; d, doublet; t, triplet; m, multiplet; br, broad) and integration. Data for ^{13}C and ^{19}F NMR are reported in terms of chemical shift (δ, ppm). IR spectra were recorded on a Perkin Elmer Spectrum Two instrument as neat samples.

High Resolution Mass Spectrometry (HRMS) data were obtained by Dr. Iain Goodall and Dr. Perry Devo of the University of Greenwich Mass Spectrometry Service using a Waters Synapt G2 hybrid Quadrupole-orthogonal acceleration time-of-flight configuration (Waters, Manchester, UK) operating in Resolution Mode (M/ΔM ≥ 18,000), fitted with a Waters Acquity UPLC binary solvent chromatographic pump system. The column used was a reversed-phase Acquity BEH C18 2.1 x 50 mm, 1.7-micron bead, running a 3- minute separation with an A:B eluent mixture comprising of either deionised water with 0.1% (v:v) formic acid and acetonitrile with 0.1% (v:v) formic acid (negative mode) respectively or deionised water with 0.1% (v:v) ammonium hydroxide and acetonitrile with 0.1% (v:v) ammonium hydroxide (positive mode) respectively. Mass calibration of the instrument was performed using sodium formate cluster ions, and an orthogonal Lock-SprayTM ESI probe was used with a lock mass calibrant, leucine-enkephalin. The pseudomolecular leucine-enkephalin ion at *m/z* = 554.2615 (Negative Ion Mode), and *m/z* = 556.2771 (Positive Ion Mode), was used as the internal mass correction calibrant. Additional samples were analyzed on a Thermo LTQ Orbitrap XL coupled with a heated electrospray source (HESI). The capillary temperature was set to 275 °C and a voltage of 21 V. The sheath gas and auxiliary gas flow were set to 10 and 5 L h^{-1} respectively and the source current and voltage set to 100 µA and 5 kV. A solution of analyte (0.1 mg/ml) and sodium formate (1% v/v) in acetonitrile was added by direct infusion (10 µL/min) into the mass spectrometer using a Hamilton syringe (250 µL).

Gas-Chromatography Mass Spectrometry (GC-MS) data were obtained using a Shimadzu Nexis GC-2030 gas chromatograph connected to a GCMS-QP2020 NX gas chromatograph mass spectrometer, equipped with an AOC-20i Plus auto injector. The column was a CD-5MS capillary column (30 m x 0.25 mm x 0.25 µm), with helium as the carrier gas. The sample injection volume was 1 µL, and separations run over a 5-minute period with an increasing oven temperature (gradient) between 40 – 280 °C. Results were visualised and manipulated using LabSolutions GCMS solution version 4.50.

High-Performance Liquid Chromatography-Mass Spectrometry (HPLC-MS) data were obtained using a Shimadzu LC-2050C 3D coupled with a Shimadzu LCMS-2020 FCV-20AH2. The column was an Ascentis Express 90Å AQ-C18, 2.7 µm. Results were visualised and manipulated using LabSolutions GCMS solution version

Cyclic voltammetry studies were carried out using an Autolab 302N potentiostat interfaced through Nova 2.1 software to a personal computer. Electrochemical measurements were performed in a glovebox under an atmosphere of dinitrogen with oxygen and water levels of less than 5 ppm at 298 K, with solvents that had been thoroughly degassed and purified by passing through an alumina-based purification system. Sample concentrations of 1.0 mM were used, alongside 0.1 M $[^nBu_4N][PF_6]$ supporting electrolyte concentrations. Experiments were conducted using a standard three-electrode setup comprising of a glassy carbon disc working electrode, platinum wire counter electrode, and AgCl coated silver wire as a pseudo-reference electrode. Potentials are reported relative to the $[FeCp_2]^{+/0}$ redox couple, obtained through the addition of ferrocene to the analyte solution.

Electrochemical Reaction Setup

Figure S1: Photograph of batch electrochemical synthesis setup (disassembled)

Cyclic Voltammetry studies

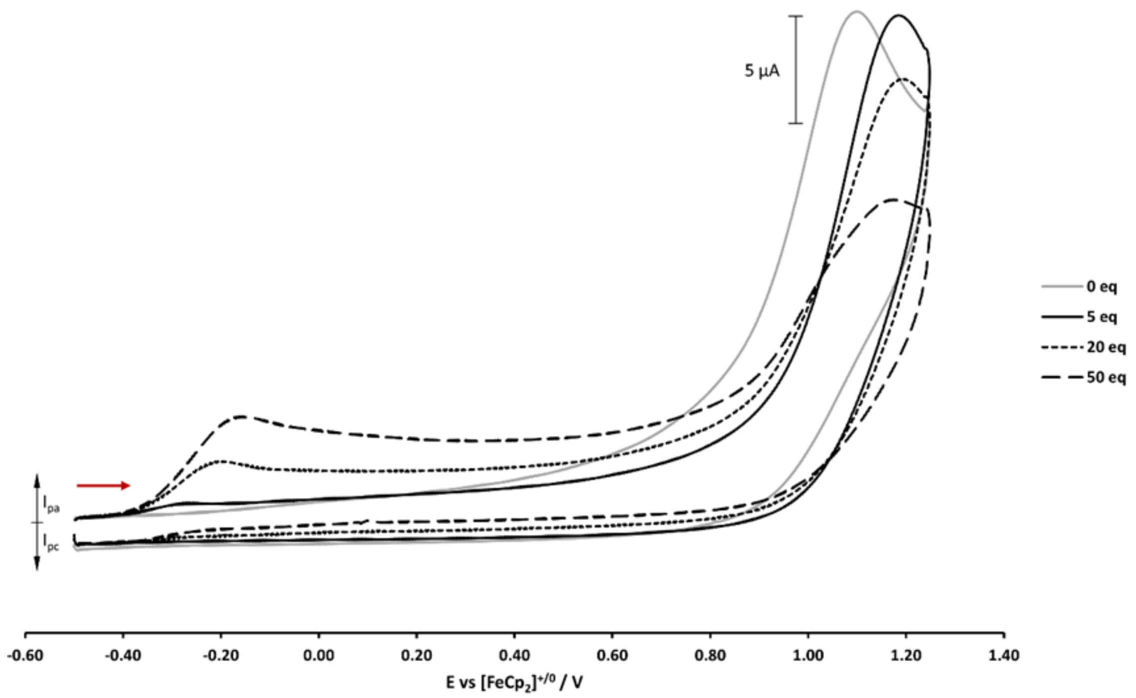


Figure S2 Cyclic voltammetry on glassy carbon (diameter 3 mm) of 1 mM solution of octylamine and 0 eq (grey line), 5 eq. (black line), 20 eq (dotted line) and 50 eq (dashed line) of CS_2 in CH_2Cl_2, 0.25V.s^{-1} scan rate.

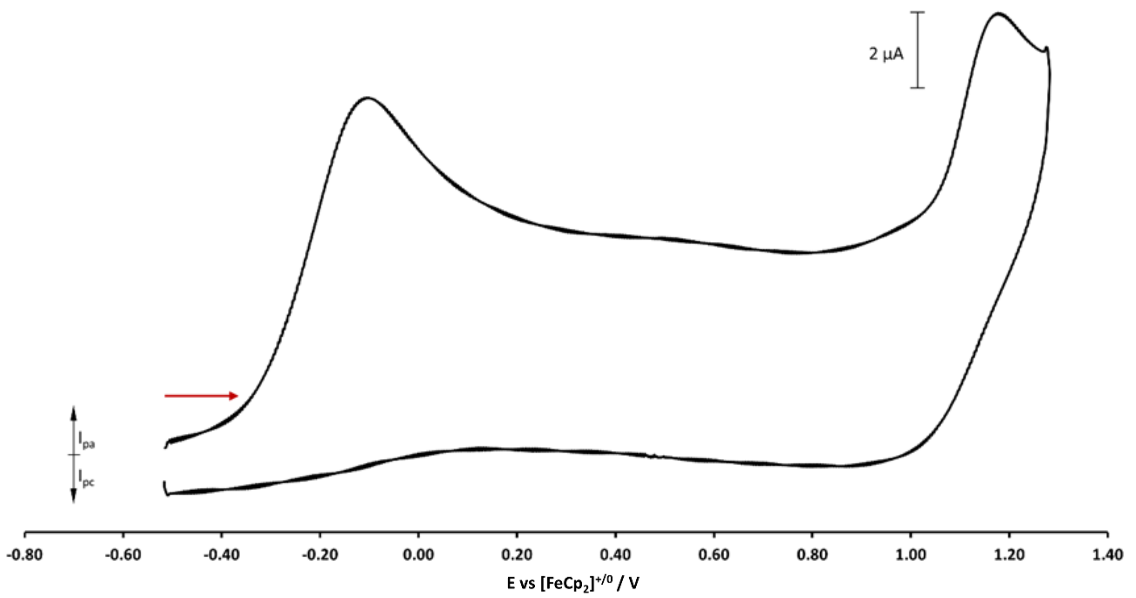
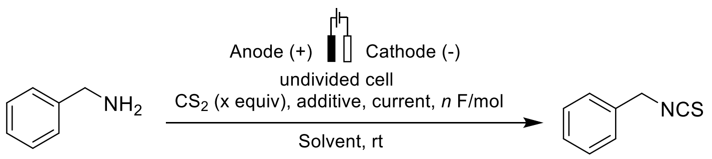


Figure S3 Cyclic voltammetry on glassy carbon (diameter 3 mm) of a solution of neat octylamine and 5 eq. of neat CS_2, 0.25V.s^{-1} scan rate.

Experimental procedures

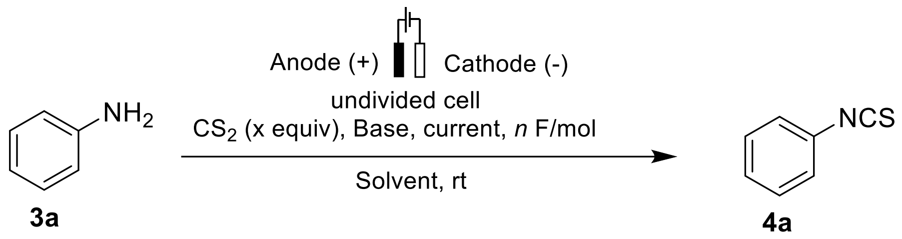
A. Summary of optimisation experiments

Table S1. Optimisation of the conversion of primary alkyl amines- [a] All yields displayed are isolated yields for **2a**. [b] Experiment done by adding CS_2 to an 80 mM solution of the amine.



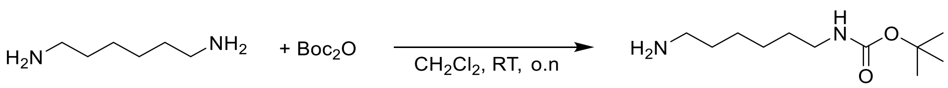
Entry	Cathode	Anode	Equivalents	Solvent	Current	F/mol	Yield[a]
1 [b]	C_{gr}	C_{gr}	1.5	MeOH	20	3	14
2 [b]	C_{gr}	C_{gr}	2.5	MeOH	20	3	22
3 [b]	C_{gr}	C_{gr}	2.5	MeOH	20	3	20
4 [b]	C_{gr}	C_{gr}	5	MeOH	20	3	20
5 [b]	C_{gr}	C_{gr}	10	MeOH	20	3	22
6	Pt	Pt	2.5	MeOH	20	3	78
7	C_{gr}	C_{gr}	2.5	MeOH	20	3	85
8	C_{gr}	C_{gr}	2.5	EtOH	20	3	41
9	C_{gr}	Ni	2.5	MeCN	20	3	80
10	C_{gr}	Ni	2.5	MeOH	50	3	12
11	C_{gr}	Ni	2.5	MeOH	10	3	82
12	C_{gr}	Ni	2.5	MeOH	5	3	84
13	C_{gr}	Ni	5	MeOH	5	3	90
14	C_{gr}	Ni	5	MeOH	5	2.2	95
15	C_{gr}	C_{gr}	5	MeOH	5	3	67

Table S2. Optimisation of the conversion of primary aryl amines

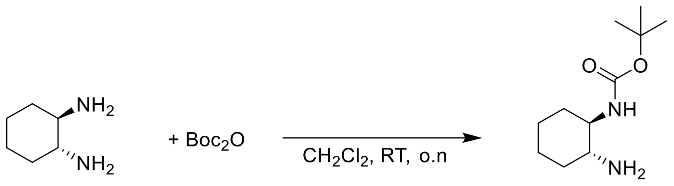


Entry	Cathode	Anode	Scale	Equiv.	Solvent	Base	Current	F/mol	Yield
1	C_{gr}	Ni	0.4 mmol	5	MeOH	Et_3N	20	3	68
2	C_{gr}	Ni	0.4 mmol	5	MeOH	Et_3N	10	3	63
3	C_{gr}	Ni	0.4 mmol	5	DCM/HFIP	DBU	20	3	52
4	C_{gr}	Ni	0.4 mmol	5	DCM/HFIP	DBU	20	5	37
5	C_{gr}	Ni	0.4 mmol	5	MeOH	-	5	3	30
6	C_{gr}	Ni	0.4 mmol	5	MeOH	Et_3N	5	3	48
7	C_{gr}	Ni	0.4 mmol	5	MeOH	Et_3N	5	2.2	20
8	C_{gr}	Ni	0.4 mmol	5	DCM	DBU	5	2.2	50
9	C_{gr}	Ni	0.4 mmol	5	DCM	Et_3N	5	2.2	45
10	C_{gr}	Ni	0.4 mmol	5	H_2O	K_2CO_3	5	2.2	0
11	C_{gr}	Ni	0.4 mmol	10	H_2O	K_2CO_3	5	2.2	0
12	C_{gr}	Ni	0.4 mmol	10	DCM	DBU	5	2.2	38
13	C_{gr}	Ni	0.4 mmol	10	DCM	Et_3N	5	2.2	48
14	C_{gr}	Ni	0.4 mmol	10	DCM/HFIP	DBU	5	2.2	52
15	C_{gr}	Ni	0.4 mmol	10	DCM/HFIP	Et_3N	5	2.2	22
16	C_{gr}	Ni	1 mmol	5	DCM 10ml	Et_3N	5	2.2	64
17	C_{gr}	Ni	1 mmol	5	DCM 10ml	DBU	5	2.2	67
18	C_{gr}	Ni	1 mmol	5	DCM	DBU	5	2.2	71

B. Synthesis of mono Boc-protected diamines

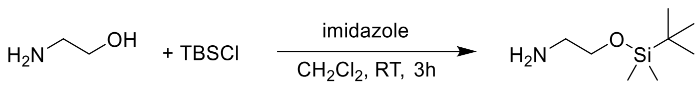


Synthesised according to literature procedure[1]. Di-*tert*-butyl dicarbonate (0.500 g, 2.29 mmol) in CH_2Cl_2 was added dropwise over a 2 h period to a 0.25 M solution of hexamethylenediamine (1.33 g, 11.5 mmol) in CH_2Cl_2 cooled with an ice bath. The reaction mixture was stirred overnight at room temperature and filtered. The filtrate was concentrated under vaccum and the resulting oil dissolved in EtOAc was washed with brine (3 x 30 mL), dried with sodium sulfate and concentrated under vaccum. The crude product was purified by flash chromatography (100% CH_2Cl_2 → 10% MeOH) and afforded the mono-protected diamine as a yellow liquid (0.490 g, 99%). Spectroscopic data matches with literature reports.[1]



Synthesised according to literature procedure[1]. Di-*tert*-butyl dicarbonate (0.500 g, 2.29 mmol) in CH_2Cl_2 was added dropwise over a 2 h period to a 0.25 M solution of trans-1,2-diaminocyclohexane (1.31 g, 11.5 mmol) in CH_2Cl_2 cooled with an ice bath. The reaction mixture was stirred overnight at room temperature and filtered. The filtrate was concentrated under vaccum and the resulting oil dissolved in EtOAc was washed with brine (3 x 30 mL), dried with sodium sulfate and concentrated under vaccum. The crude product was purified by flash chromatography (100% CH_2Cl_2 → 10% MeOH) and afforded the mono-protected diamine as a white solid (0.487 g, 99%). Spectroscopic data matches with literature reports.[2]

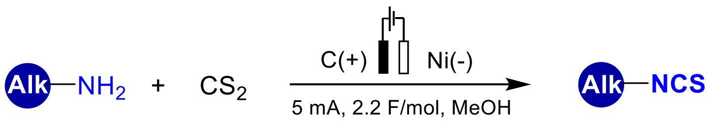
C. Synthesis of 2-((*tert*-butyldimethylsilyl)oxy)ethan-1-amine



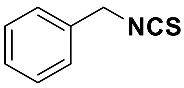
Synthesised according to literature procedure.[3] To *tert*-butylchlorodimethylsilane (TBSCl, 2.71g, 18 mmol) in CH_2Cl_2 (5 mL) was added to a mixture of ethanolamine (1.00g, 16.4 mmol) and imidazole (2.23g, 32.7 mmol) in CH_2Cl_2 (33 mL) in room temperature. The mixture was reacted for 3h and then poured into water (60 mL) and extracted with DCM (3 x 30 mL). The combined organic layers were washed with brine, dried over sodium sulfate, filtered and the filtrate was concentrated under reduced pressure. The residue was then brought to high vaccum affording 2-((*tert*-butyldimethylsilyl)oxy)ethan-1-amine as a yellow liquid (2.35g, 82%). Spectroscopic data matches with literature reports.[4]

D. Electrochemical conversion of primary amines to isothiocyanates

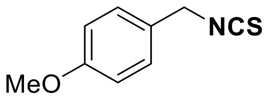
General procedure A. Conversion of primary alkyl amines to alkyl isothiocyanate



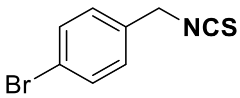
A 5 mL IKA Electrasyn electrochemical cell was charged with the alkyl amine (0.40 mmol). Carbon disulfide (CS_2, 2 mmol) was added then the mixture was dissolved in MeOH (5 mL). The mixture was then electrolysed at a constant current of 5 mA for 2.2F/mol. After reaching completion, the reaction mixture was poured in aqueous HCl (1.0M, 30 mL) and extracted with hexane (3 x 20 mL). The combined organic layers were dried over $MgSO_4$, filtered and the solvent was removed under reduced pressure affording the clean alkyl isothiocyanate.



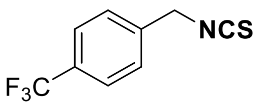
Synthesised according to general procedure A from benzylamine (0.0429 g, 0.40 mmol). Evaporation of solvent afforded isothiocyanate **2a** (0.0567 g, 95%) as a yellow oil. Isothiocyanate **2a**: ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 7.42-7.31 (m, 5H), 4.72 (s, 2H); ^{13}C {^{1}H} NMR (101 MHz, $CDCl_3$): δ_C 134.0, 131.9, 128.8, 128.2, 126.7, 48.5; IR (υ, cm^{-1}, neat): 2063 [N=C=S] ; HRMS (ESI) m/z: [M-H]$^-$ calcd for C_8H_6NS: 148.0226; found: 148.0245



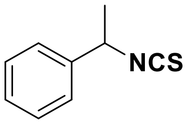
Synthesised according to general procedure A from 4-methoxybenzylamine (0.0549 g, 0.40 mmol). Evaporation of solvent afforded isothiocyanate **2b** (0.0695 g, 97%) as a yellow oil. Isothiocyanate **2b**: ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 7.24-7.23 (m, 2H), 6.92-6.89 (m, 2H), 4.63 (s, 2H), 3.81 (s, 3H); ^{13}C {^{1}H} NMR (101 MHz, $CDCl_3$): δ_C 159.7, 131.9, 128.5, 126.4, 114.4, 55.4, 48.3; IR (υ, cm^{-1}, neat): 2074 [N=C=S] ; HRMS (ESI) m/z: [M-H]$^-$ calcd for C_9H_8NOS: 178.0332; found: 178.0327



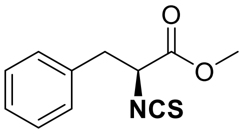
Synthesised according to general procedure A from 4-bromobenzylamine (0.0744 g, 0.40 mmol). Evaporation of solvent afforded isothiocyanate **2c** (0.0867 g, 95%) as a yellow oil. Isothiocyanate **2c**: ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 7.53-7.50 (m, 2H), 7.21-7.17 (m, 2H), 4.67 (s, 2H); ^{13}C {^{1}H} NMR (101 MHz, $CDCl_3$): δ_C 133.4, 133.1, 132.2, 128.6, 122.5, 48.2; IR (υ, cm^{-1}, neat): 2074 [N=C=S] ; HRMS (ESI) m/z: [M-H]$^-$ calcd for C_8H_5BrNS: 225.9332; found: 225.9326



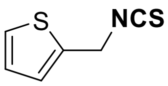
Synthesised according to general procedure A from 4-(trifluoromethyl) benzylamine (0.0701 g, 0.40 mmol). Evaporation of solvent afforded isothiocyanate **2d** (0.0851 g, 98%) as a yellow oil. Isothiocyanate **2d**: ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 7.67-7.65 (d, J = 8.11 Hz, 2H), 7.46-7.44 (d, J = 8.11 Hz, 2H), 4.80 (s, 2H); ^{13}C {^{1}H} NMR (101 MHz, $CDCl_3$): δ_C 138.3, 133.8, 130.8, 127.2, 126.0, 124.0, 48.3; ^{19}F NMR (376 MHz, $CDCl_3$): δ_F -62.53; IR (υ, cm^{-1}, neat): 1322 [C-F], 2076 [N=C=S] ; HRMS (ESI) m/z: [M+NH_4]$^+$ calcd for C9H13N_2OS: 235.0511; found: 235.0517



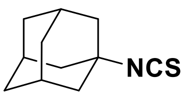
Synthesised according to general procedure A from 1-phenylethylamine (0.0484 g, 0.40 mmol). Evaporation of solvent afforded isothiocyanate **2e** (0.0620 g, 95%) as a yellow oil. Isothiocyanate **2e**: ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 7.42-7.32 (m, 5H), 4.95-4.90 (q, J = 6.79 Hz, 1H), 1.69-1.67 (d, J = 6.87 Hz, 3H); ^{13}C {^{1}H} NMR (101 MHz, $CDCl_3$): δ_C 140.2, 132.2, 129.0, 128.3, 125.5, 57.1, 25.1; IR (υ, cm^{-1}, neat): 2081 [N=C=S] ; HRMS (ESI) m/z: $[M-H]^-$ calcd for C_9H_8NS: 162.0383; found: 162.0384



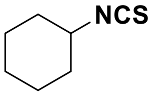
Synthesised according to general procedure A from L-phenylalanine methyl ester (0.0717 g, 0.40 mmol). Evaporation of solvent afforded isothiocyanate **2f** (0.06904 g, 78%) as a yellow oil. Isothiocyanate **2f**: ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 7.37-7.30 (m, 3H), 7.24-7.21 (dd, J = 8.23, 1.82 Hz, 2H), 4.50-4.47 (dd, J = 8.42, 4,71 Hz, 1H), 3.79 (s, 3H), 3.28-3.23 (dd, J = 13.79, 4.64 Hz, 1H), 3.16-3.10 (dd, J = 13.97, 8.35 Hz, 1H); ^{13}C {^{1}H} NMR (101 MHz, $CDCl_3$): δ_C 168.5, 137.9, 135.1, 129.4, 128.9, 127.8, 60.9, 53.3, 39.8; IR (υ, cm^{-1}, neat): 1744 [C=O], 2050 [N=C=S] ; HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{11}H_{12}NO_2S$: 222.0583; found: 222.0586



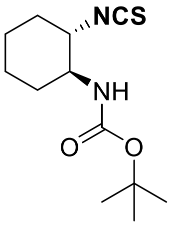
Synthesised according to general procedure A from 2-thiophenemethylamine (0.0453 g, 0.40 mmol). Evaporation of solvent afforded isothiocyanate **2g** (0.0192 g, 0.124 mmol, 31%) as a brown solid. Isothiocyanate **2g**: ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 7.30-7.29 (dd, J = 5.18 ,1.33 Hz, 1H), 7.05-7.03 (m, 1H), 6.99-6.97 (m, 1H) 4.84 (s, 2H); ^{13}C {^{1}H} NMR (101 MHz, $CDCl_3$): δ_C 136.6, 134.3, 127.2, 126.8, 126.4, 43.9; IR (υ, cm^{-1}, neat): 2069 [N=C=S] ; HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_6H_6NS_2$: 155.9936; found: 155.9926



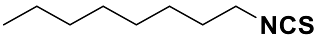
Synthesised according to general procedure A from adamantanylamine (0.0605 g, 0.40 mmol). Evaporation of solvent afforded isothiocyanate **2h** (0.0472 g, 78%) as a yellow oil. Isothiocyanate **2h**: ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 2.10-2.09 (m, 3H), 1.97-1.92 (m, 6H), 1.67-1.61 (m, 6H); ^{13}C {^{1}H} NMR (101 MHz, $CDCl_3$): δ_C 129.4, 58.5, 43.8, 35.6, 29.3; IR (υ, cm^{-1}, neat): 2056 [N=C=S] ; HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{11}H_{16}NS$: 193.0920; found: 193.0924



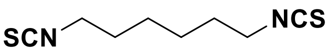
Synthesised according to general procedure A from cyclohexylamine (0.0397 g, 0.40 mmol). Evaporation of solvent afforded isothiocyanate **2i** (0.0365 g, 92%) as a yellow oil. Isothiocyanate **2i**: ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 3.71-3.65 (m, 1H), 1.90-1.86 (m, 2H), 1.72-1.59 (m, 4H), 1.51-1.45 (m, 1H), 1.39-1.33 (m, 3H); ^{13}C {^{1}H} NMR (101 MHz, $CDCl_3$): δ_C 129.6, 55.5, 33.3, 25.1, 23.3; IR (υ, cm^{-1}, neat): 2093 [N=C=S] ; HRMS (ESI) m/z: [M+H]$^{-}$ calcd for $C_7H_{12}NS$: 142.0685; found: 142.0695



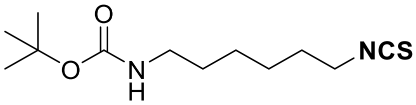
Synthesised according to general procedure A from *tert*-butyl((1*S*,2*S*)-2-aminocyclohexyl) carbamate (0.0857 g, 0.40 mmol). Evaporation of solvent afforded isothiocyanate **2j** (0.0301 g, 29%) as a white solid. Isothiocyanate **2j**: ^{1}H NMR (500 MHz, $CDCl_3$): δ_H 4.58 (s, 1H), 3.52 (d, J = 32Hz, 2H), 2.18 – 1.93 (m, 2H), 1.76 – 1.64 (m, 2H), 1.59 (m, 1H), 1.46 (s, 9H), 1.42 – 1.18 (m, 4H) ; ^{13}C {^{1}H} NMR (126 MHz, $CDCl_3$): δ_C 155.2, 132.4, 80.2, 60.5, 53.9, 32.4, 31.6, 28.6, 24.1, 23.6 ; IR (υ, cm^{-1}, neat): 1678 [C=O], 2098 [N=C=S] ; Spectroscopic data matches with literature reports.[5]



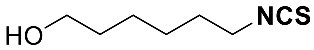
Synthesised according to general procedure A from octylamine (0.0517 g, 0.40 mmol). Evaporation of solvent afforded isothiocyanate **2k** (0.0512 g, 99%) as a yellow oil. Isothiocyanate **2k**: ^{1}H NMR (500 MHz, $CDCl_3$): δ_H 3.51-3.48 (t, J = 7.12 Hz, 2H), 1.70-1.65 (m, 2H), 1.41-1.36 (m, 2H), 1.29-1.25 (m, 8H), 0.88-0.85 (t, J = 6.98 Hz, 3H); ^{13}C {^{1}H} NMR (126 MHz, $CDCl_3$): δ_C 129.5, 45.1, 31.8, 30.0, 29.1, 28.8, 26.6, 22.7, 14.2; IR (υ, cm^{-1}, neat): Spectroscopic data matches with literature report.[6] ; HRMS (ESI) m/z: [M+H]$^{+}$ calcd for $C_9H_{18}NS$: 172.1155; found: 172.1155



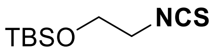
Synthesised according to general procedure A with 10 equiv. CS_2 and electrolysed with a constant current of 5 mA for 4.4F/mol from hexamethylenediamine (0.0465 g, 0.40 mmol). Evaporation of solvent afforded isothiocyanate **2l** (0.0330 g, 71%) as a yellow oil. Isothiocyanate **2l**: ^{1}H NMR (500 MHz, $CDCl_3$): δ_H 3.55-3.31 (t, J = 6.51 Hz, 2H), 1.76-1.68 (m, 2H), 1.47-1.44 (m, 2H); ^{13}C {^{1}H} NMR (126 MHz, $CDCl_3$): δ_C 130.0, 45.0, 29.8, 26.0; IR (υ, cm^{-1}, neat): 2081 [N=C=S]; HRMS (ESI) m/z: [M+H]$^{+}$ calcd for $C_8H_{13}N_2S_2$: 201.0515; found: 201.0522



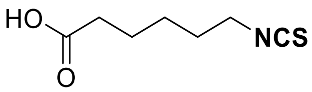
Synthesised according to general procedure A from *tert*-butyl (6-aminohexyl) carbamate (0.0865 g, 0.40 mmol). Evaporation of solvent afforded isothiocyanate **2m** (0.0548 g, 53%) as a yellow oil. Isothiocyanate **2m**: ^{1}H NMR (500 MHz, $CDCl_3$): δ_H 4.53 (s, 1H), 3.50 (t, *J* = 6.6 Hz, 2H), 3.11 (m, 2H), 1.74 – 1.62 (m, 2H), 1.53 – 1.38 (m, 14H), 1.37 – 1.29 (m, 2H) ; ^{13}C {^{1}H} NMR (126 MHz, $CDCl_3$): δ_C 156.1, 129.8, 79.3, 45.1, 40.5, 30.1, 30.0, 28.5, 26.4, 26.1; IR (υ, cm^{-1}, neat): 1689 [C=O], 2093 [N=C=S] ; HRMS (ESI) m/z [M+H]$^+$ calcd for $C_{12}H_{23}N_2O_2S$: 259.1480; found: 259.1480



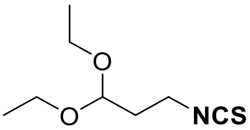
Synthesised according to general procedure A from 6-amino-1-hexanol (0.0469 g, 0.40 mmol). Evaporation of solvent afforded isothiocyanate **2n** (0.0347 g, 74%) as a yellow oil. Isothiocyanate **2n**: ^{1}H NMR (500 MHz, $CDCl_3$): δ_H 3.65-3.62 (t, J = 6.58 Hz, 2H), 3.53-3.49 (t, J = 6.58 Hz, 2H), 1.72-1.68 (m, 3H), 1.59-1.55 (m, 2H), 1.44-1.38 (m, 4H); ^{13}C {^{1}H} NMR (126 MHz, $CDCl_3$): δ_C 129.5, 62.7, 45.0, 32.5, 30.0, 26.4, 25.1; IR (υ, cm^{-1}, neat): 2087 [N=C=S], 3332 [O-H] ; HRMS (ESI) m/z: [M+H]$^+$ calcd for $C_7H_{14}NOS$: 160.0791; found: 160.0784



Synthesised according to general procedure A from 2-((*tert*-butyldimethylsilyl)oxy)ethan-1-amine (0.0701 g, 0.40 mmol). Evaporation of solvent afforded isothiocyanate **2o** (0.0537 g, 62%) as a colourless solid. Isothiocyanate **2o**: ^{1}H NMR (500 MHz, $CDCl_3$): δ_H 3.80 (t, *J* = 5.4 Hz, 2H), 3.57 (t, *J* = 5.4 Hz, 2H), 0.91 (s, 9H), 0.10 (s, 6H); ^{13}C {^{1}H} NMR (126 MHz, $CDCl_3$): δ_C 132.4, 61.8, 47.8, 25.8, 18.3, -5.3; IR (υ, cm^{-1}, neat): 2087 [N=C=S] ; HRMS (ESI) m/z: [M+H]$^+$ calcd for $C_9H_{20}NOSSi$: 218.1035; found: 218.1049

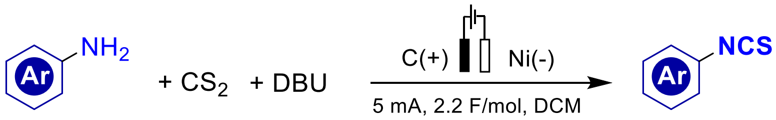


Synthesised according to general procedure A from 6-aminohexanoic acid (0.0525 g, 0.40 mmol). Evaporation of solvent afforded isothiocyanate **2p** (0.0686 g, 99%) as a yellow oil. Isothiocyanate **2p**: ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 3.54-3.51 (t, J = 7.40 Hz, 2H), 2.40-2.37 (t. J = 7.40 Hz, 2H), 1.75-1.64 (m, 4H), 1.51-1.44 (m, 2H); ^{13}C {^{1}H} NMR (101 MHz, $CDCl_3$): δ_C 179.7, 130.3, 45.0, 33.8, 29.8, 26.1, 23.9; IR (υ, cm^{-1}, neat): 1705 [C=O], 2090 [N=C=S] ; HRMS (ESI) m/z: [M-H]$^+$ calcd for $C_7H_{10}NO_2S$: 172.0427; found: 172.0420

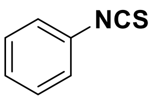


Synthesised according to general procedure A from 1-amino-3,3-diethoxypropane (0.0589 g, 0.40 mmol). Evaporation of solvent afforded isothiocyanate **2q** (0.0300 g, 40%) as a dark green oil. Isothiocyanate **2q**: ^{1}H NMR (500 MHz , $CDCl_3$): δ_H 4.60 (m, 1H), 3.71 – 3.57 (m, 4H), 3.50 (m, 2H), 1.96 (m, 2H), 1.20 (m, 6H) ; ^{13}C {^{1}H} NMR (126 MHz , $CDCl_3$): δ_C 130.3, 100.2, 62.4, 41.3, 34.2, 15.4; IR (υ, cm^{-1}, neat): 2093 [N=C=S] Spectroscopic data matches with literature reports.[7]

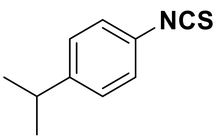
General procedure B. Conversion of primary aryl amines to aryl isothiocyanate



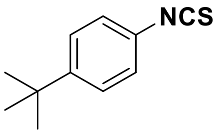
A 5 mL IKA Electrasyn electrochemical cell was charged with the aryl amine (1 mmol). Carbon disulfide (CS_2, 2 mmol) and DBU (2 mmol) was added, the mixture was stirred for 5 min before being dissolved in DCM (5 mL). The mixture was then electrolysed at a constant current of 5 mA for 2.2F/mol. After reaching completion, the reaction mixture was poured in aqueous HCl (1.0M, 30 mL) and extracted with hexane (3 x 20 mL). The combined organic layers were dried over $MgSO_4$, filtered and the solvent was removed under reduced pressure. Filtration over silica gel with hexane was performed when necessary. The solvent was removed under reduced pressure affording the clean aryl isothiocyanate.



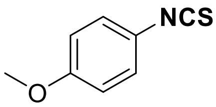
Synthesised according to general procedure B from aniline (0.0372 g, 0.40 mmol). Purification through SiO_2 with hexane as solvent afforded isothiocyanate **4a** (0.0384 g, 71%) as a yellow oil. Isothiocyanate **4a**: ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 7.35-7.31 (m, 2H), 7.28-7.24 (m, 1H), 7.21-7.18 (m, 2H); ^{13}C {^{1}H} NMR (101 MHz, $CDCl_3$): δ_C 135.3, 131.2, 129.6, 127.4, 125.8; IR (υ, cm^{-1}, neat): 2041 [N=C=S] ; HRMS (ESI) m/z: $[M-H]^-$ calcd for C_7H_4NS: 134.0070; found: 134.0091



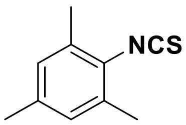
Synthesised according to general procedure B from 4-isopropylaniline (0.0541 g, 0.40 mmol). Purification through SiO_2 with hexane as solvent afforded isothiocyanate **4b** (0.0674 g, 0.38 mmol, 95%) as a yellow oil. Isothiocyanate **4b**: ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 7.32-7.18 (m, 4H), 3.31-3.25 (m, 1H), 1.30-1.29 (d, J = 6.92 Hz, 6H); ^{13}C {^{1}H} NMR (101 MHz, $CDCl_3$): δ_C 144.7, 135.3, 129.4, 127.8, 126.9, 126.9, 126.4, 29.7, 22.9; IR (υ, cm^{-1}, neat): 2077 [N=C=S] ; HRMS (ESI) m/z: $[M+NH_4]^+$ calcd for $C_{10}H_{15}N_2S$: 195.0950; found: 195.0945



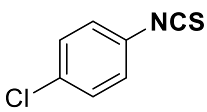
Synthesised according to general procedure B from 4-*tert*-butylaniline (0.0597 g, 0.40 mmol). Purification through SiO_2 with hexane as solvent afforded isothiocyanate **4c** (0.0712 g, 93%) as a brown solid. Isothiocyanate **4c**: ^1H NMR (400 MHz, $CDCl_3$): δ_H 7.38-7.34 (m, 2H), 7.17-7.14 (m, 2H), 1.31 (s, 9H); ^{13}C {^1H} NMR (101 MHz, $CDCl_3$): δ_C 150.8, 134.5, 128.4, 126.5, 125.4, 34.8, 31.3; IR (υ, cm^{-1}, neat): 2050 [N=C=S] ; HRMS (ESI) m/z: $[M+Na]^+$ calcd for $C_{11}H_{13}NSNa$: 214.0661; found: 214.0641



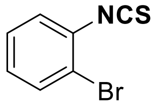
Synthesised according to general procedure B from 4-methoxyaniline (0.0493 g, 0.40 mmol). Purification through SiO_2 with hexane as solvent afforded isothiocyanate **4d** (0.0529 g, 80%) as a yellow oil. Isothiocyanate **4d**: ^1H NMR (400 MHz, $CDCl_3$): δ_H 7.15-7.12 (m, 2H), 6.85-6.82 (m, 2H), 3.78 (s, 3H); ^{13}C {^1H} NMR (101 MHz, $CDCl_3$): δ_C 158.6, 133.8, 127.0, 123.5, 114.8, 55.6; IR (υ, cm^{-1}, neat): 2050 [N=C=S] ; HRMS (ESI) m/z: $[M+MeOH+H]^+$ calcd for $C_9H_{12}NO_2S$: 198.0583; found: 198.0589



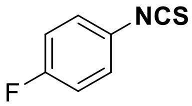
Synthesised according to general procedure B from 2,4,6-trimethylaniline (0.0541 g, 0.40 mmol). Purification through SiO_2 with hexane as solvent afforded isothiocyanate **4e** (0.0581 g, 82%) as a white solid. Isothiocyanate **4e**: ^1H NMR (400 MHz, $CDCl_3$): δ_H 6.85 (s, 1H), 2.33 (s, 6H), 2.28 (s, 3H); ^{13}C {^1H} NMR (101 MHz, $CDCl_3$): δ_C 137.1, 134.9, 134.9, 128.8, 126.9, 21.1, 18.6; IR (υ, cm^{-1}, neat): 2105 [N=C=S] ; HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_{10}H_{12}NS$: 178.0685; found: 178.0692



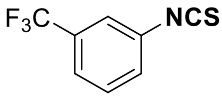
Synthesised according to general procedure B from 4-chloroaniline (0.0510 g, 0.40 mmol). Purification through SiO_2 with hexane as solvent afforded isothiocyanate **4f** (0.0271 g, 40%) as a yellow oil. Isothiocyanate **4f**: ^1H NMR (400 MHz, $CDCl_3$): δ_H 7.32-7.30 (m, 2H), 7.16-7.13 (m, 2H); ^{13}C {^1H} NMR (101 MHz, $CDCl_3$): δ_C 136.8, 133.0, 130.0, 129.9, 127.0; IR (υ, cm^{-1}, neat): 2075 [N=C=S] ; HRMS (ESI) m/z: $[M+MeO]^-$ calcd for C_8H_7OClNS: 199.9942; found: 199.9937



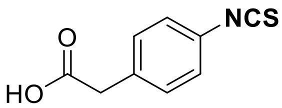
Synthesised according to general procedure B from 2-bromoaniline (0.0688 g, 0.40 mmol). Purification through SiO_2 with hexane as solvent afforded isothiocyanate **4g** (0.0788 g, 92%) as a yellow solid. Isothiocyanate **4g**: ^1H NMR (400 MHz, $CDCl_3$): δ_H 7.56-7.54 (dd, J = 8.16, 1.36 Hz, 1H), 7.27-7.21 (m, 2H), 7.11-7.08 (m, 1H); ^{13}C {^1H} NMR (101 MHz, $CDCl_3$): δ_C 138.1, 133.5, 131.6, 128.4, 128.3, 127.2, 120.9; IR (υ, cm^{-1}, neat): 2038 [N=C=S] ; HRMS (ESI) m/z: $[M+H]^+$ calcd for C_7H_5BrNS: 213.9321; found: 213.9330



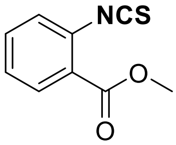
Synthesised according to general procedure B from 4-fluoroaniline (0.0444 g, 0.40 mmol). Purification through SiO_2 with hexane as solvent afforded isothiocyanate **4h** (0.0539 g, 88%) as a brown solid. Isothiocyanate **4h**: ^1H NMR (400 MHz, $CDCl_3$): δ_H 7.23-7.19 (m, 2H), 7.07-7.02 (m, 2H); ^{13}C {^1H} NMR (101 MHz, $CDCl_3$): δ_C 162.5, 160.0, 135.9, 127.6, 116.9; ^{19}F-NMR (376 MHz, $CDCl_3$) δ_F -111.92; IR (υ, cm^{-1}, neat): 1499 [C-F], 2050 [N=C=S] ; HRMS (ESI) m/z: $[M+H]^+$ calcd for C_7H_5FNS: 154.0121; found: 154.0122



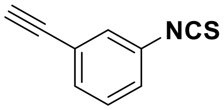
Synthesised according to general procedure B from 3-trifluoromethylaniline (0.0644 g, 0.40 mmol). Purification through SiO_2 with hexane as solvent afforded isothiocyanate **4i** (0.0512 g, 63%) as a yellow solid. Isothiocyanate **4i**: ^1H NMR (400 MHz, $CDCl_3$): δ_H 7.54-7.47 (m, 3H), 7.41-7.39 (m, 1H); ^{13}C {^1H} NMR (101 MHz, $CDCl_3$): δ_C 138.4, 132.6, 132.5, 130.4, 129.0, 123.9, 123.4, 122.8; ^{19}F-NMR (376 MHz, $CDCl_3$) δ_F -62.89; IR (υ, cm^{-1}, neat): 1329 [C-F], 2049 [N=C=S] ; HRMS (ESI) m/z: $[M+H]^-$ calcd for $C_8H_5F_3NS$: 204.0100; found: 204.0101



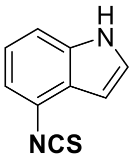
Synthesised according to general procedure B from 2-(4-aminophenyl) acetic acid (0.0605 g, 0.40 mmol). Purification through SiO_2 with hexane as solvent afforded isothiocyanate **4j** (0.0641 g, 83%) as a yellow oil. Isothiocyanate **4j**: ^1H NMR (400 MHz, $CDCl_3$): δ_H 7.28-7.25 (m, 2H), 7.20-7.18 (m, 2H), 3.65 (s, 2H); ^{13}C {^1H} NMR (101 MHz, $CDCl_3$): δ_C 177.1, 136.0, 132.6, 130.8, 130.7, 126.1, 40.6; IR (υ, cm^{-1}, neat): 1692 [C=O], 2093 [N=C=S] ; HRMS (ESI) m/z: $[M-H]^+$ calcd for $C_9H_6NO_2S$: 192.0114; found: 192.0107



Synthesised according to general procedure B from methyl-2-aminobenzoate (0.0605 g, 0.40 mmol). Purification through SiO_2 with hexane as solvent afforded isothiocyanate **4k** (0.0525 g, 68%) as a yellow solid. Isothiocyanate **4k**: ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 7.99-7.97 (dd, J = 7.91, 1.55 Hz, 1H), 7.52-7.49 (m, 1H), 7.35-7.28 (m, 2H), 3.97 (s, 3H); ^{13}C {^{1}H} NMR (101 MHz, $CDCl_3$): δ_C 165.2, 135.9, 133.4, 131.9, 130.6, 127.8, 127.0, 126.5, 52.9; IR (υ, cm^{-1}, neat): 1719 [C=O], 2089 [N=C=S] ; HRMS (ESI) m/z: $[M+H]^+$ calcd for $C_9H_8NO_2S$: 194.0270; found: 194.0269



Synthesised according to general procedure B from 3-ethynylaniline (0.0469 g, 0.40 mmol). Purification through SiO_2 with hexane as solvent afforded isothiocyanate **4l** (0.0280 g, 44%) as a yellow oil. Isothiocyanate **4l**: ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 7.39-7.36 (m, 1H), 7.33-7.32 (m, 1H), 7.30-7.28 (m, 1H), 7.21-7.18 (m, 1H), 3.13 (s, 1H); ^{13}C {^{1}H} NMR (101 MHz, $CDCl_3$): δ_C 136.8, 131.7, 131.0, 129.7, 129.2, 126.2, 123.9, 82.0, 79.0; IR (υ, cm^{-1}, neat): 2033 [N=C=S] ; HRMS (ESI) m/z: $[M-H]^-$ calcd for C_9H_4NS: 158.0070; found: 158.0069



Synthesised according to general procedure B from 4-aminoindole (0.0529 g, 0.40 mmol). Purification through SiO_2 with hexane as solvent afforded isothiocyanate **30** (0.0118 g, 17%) as a yellow oil. isothiocyanate **30**: ^{1}H NMR (400 MHz, $CDCl_3$): δ_H 8.29 (s, 1H), 7.29-7.26 (m, 1H), 7.21-7.20 (t, J = 2.97 Hz, 1H), 7.08-7.04 (t, J = 7.96 Hz, 1H), 6.93-6.90 (m, 1H), 6.66-6.65 (m, 1H); ^{13}C {^{1}H} NMR (101 MHz, $CDCl_3$): δ_C 136.7, 135.9, 125.4, 125.3, 122.9, 122.3, 116.8, 111.0, 100.4; IR (υ, cm^{-1}, neat): 3419 [N-H], 2052 [N=C=S] ; HRMS (ESI) m/z: $[M-H]^-$ calcd for $C_9H_5N_2S$: 173.0179; found: 173.0173

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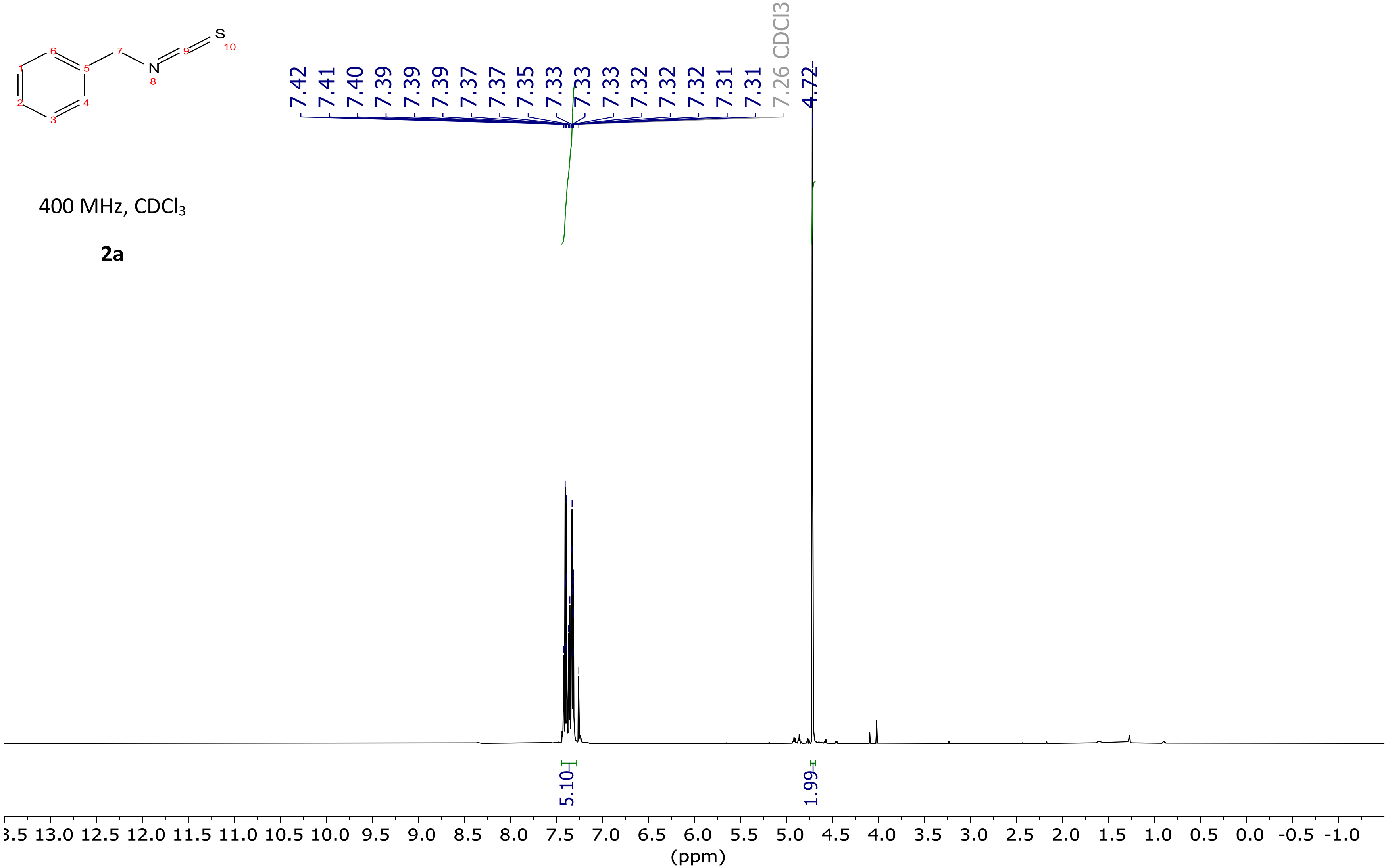
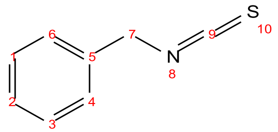
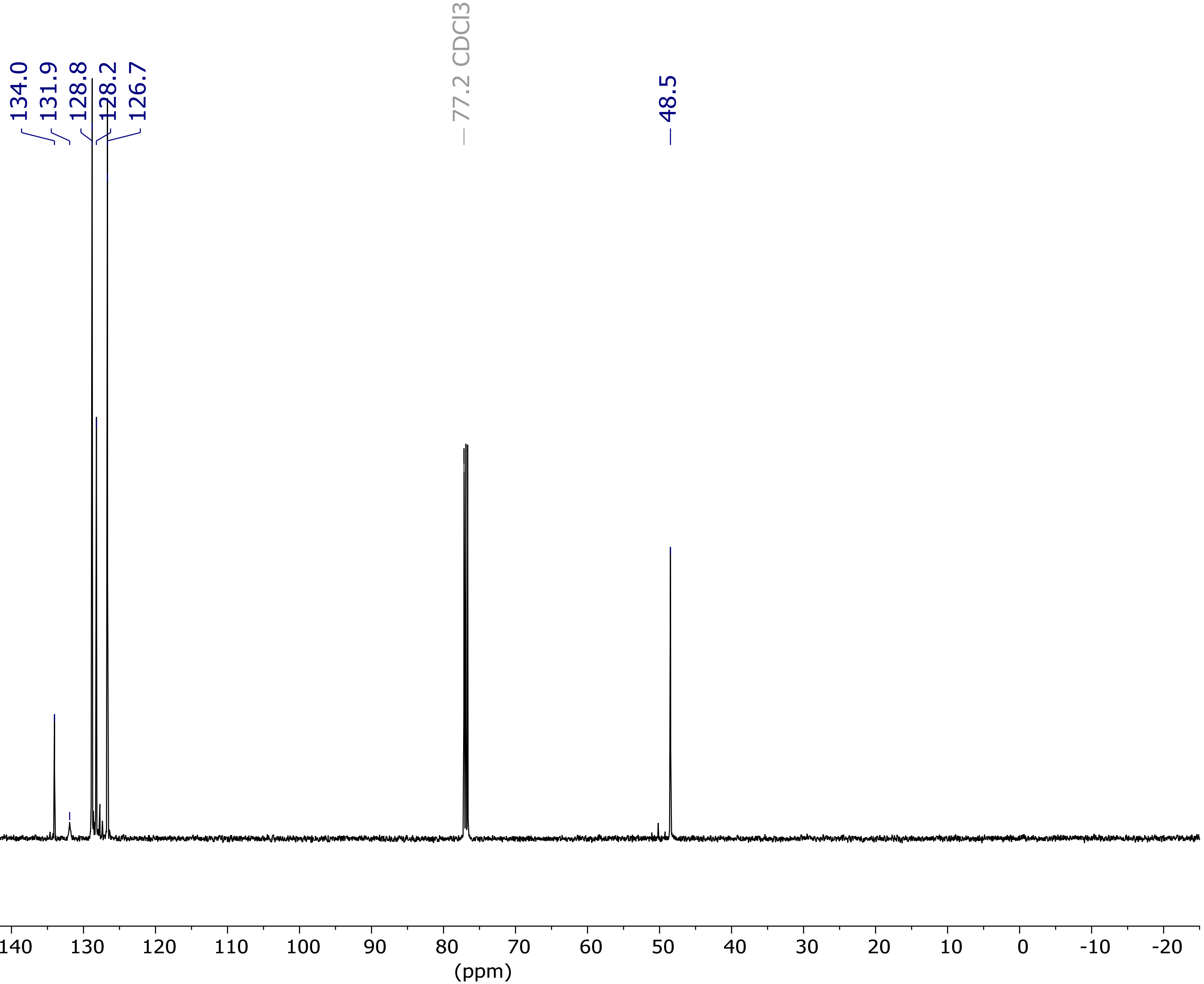
NMR Spectra

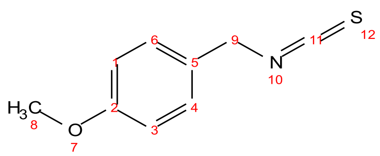

S
10
N
8
400 MHz, CDCl₃
2a
7.42 7.41 7.40 7.39 7.39 7.39 7.37 7.37 7.35 7.33 7.33 7.33 7.32 7.32 7.32 7.31 7.31
7.26 CDCl3
4.72
5.10
1.99
(ppm)



101 MHz, $CDCl_3$

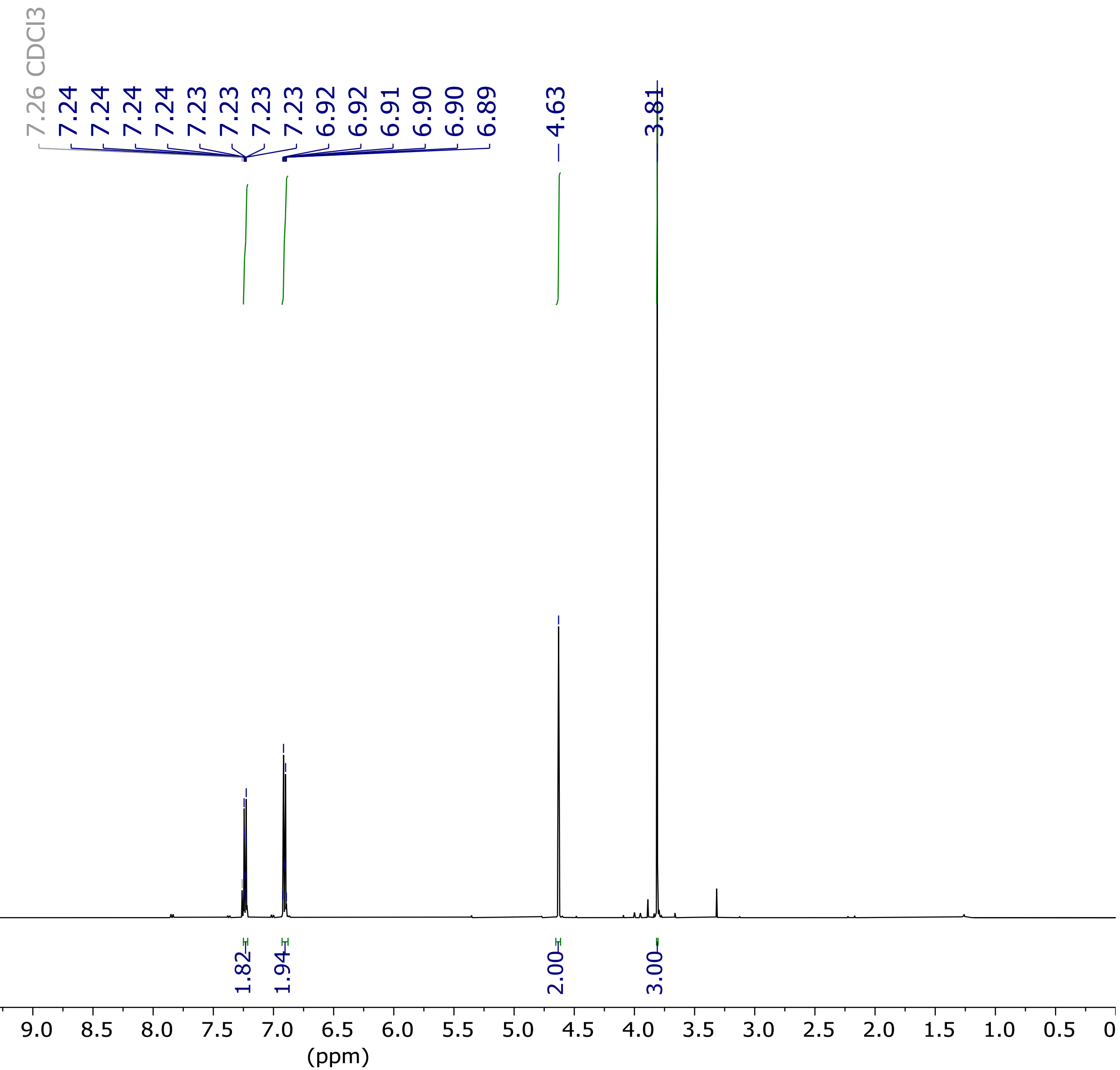
2a

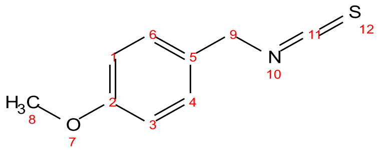




400 MHz, $CDCl_3$

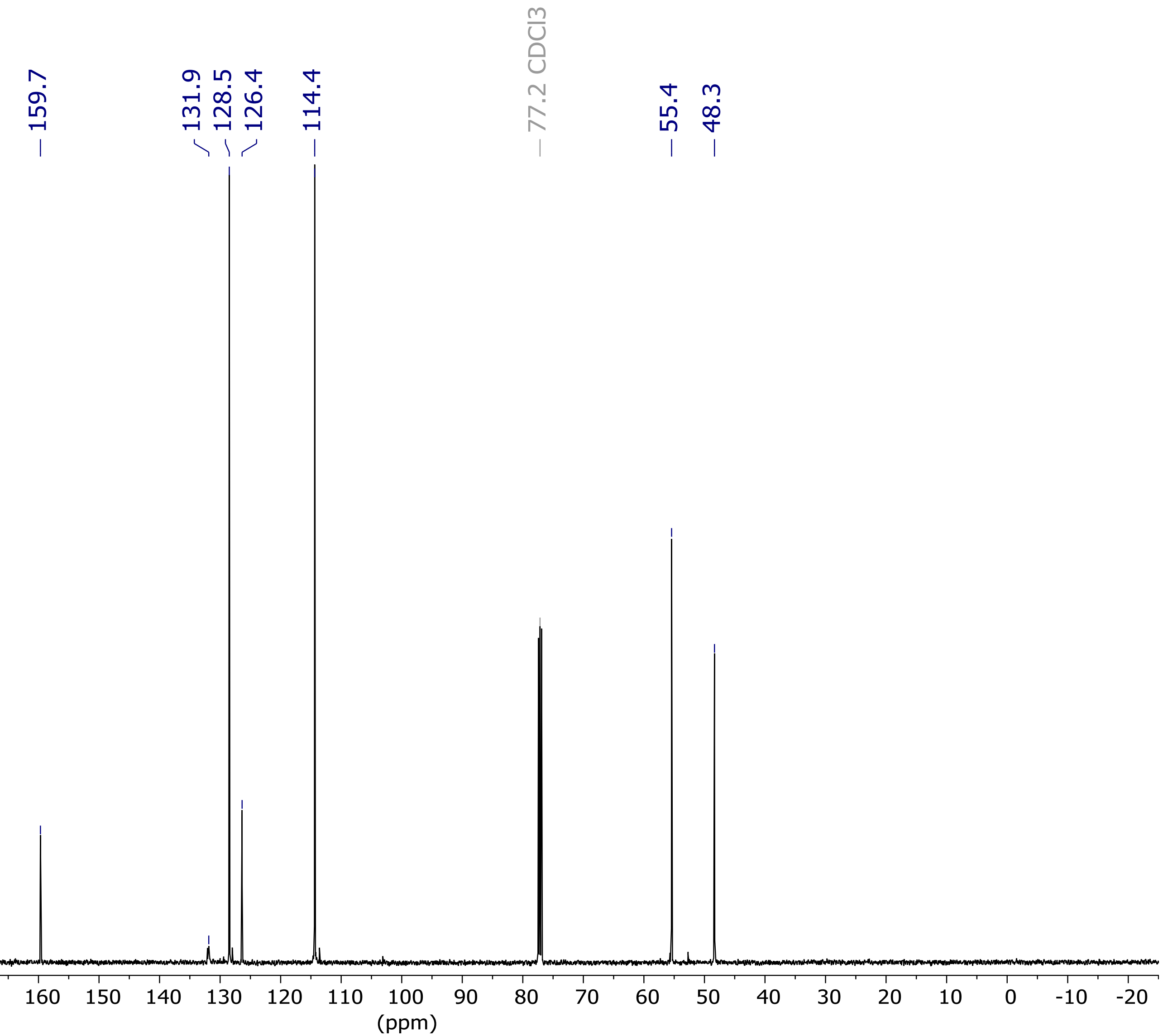
2b

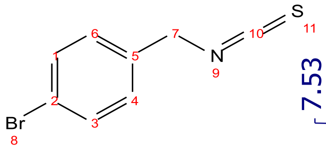




101 MHz, $CDCl_3$

2b





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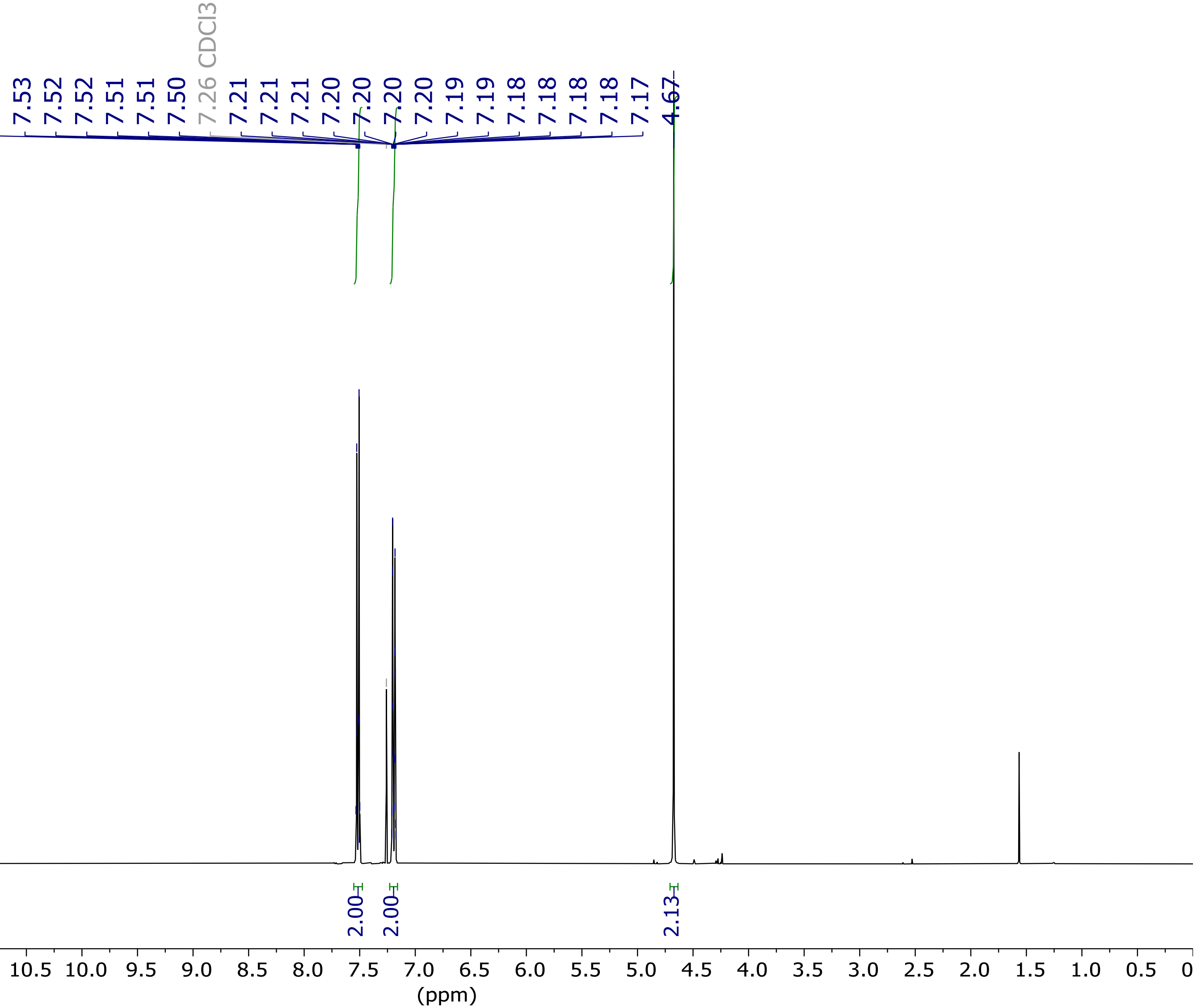
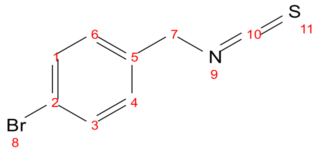
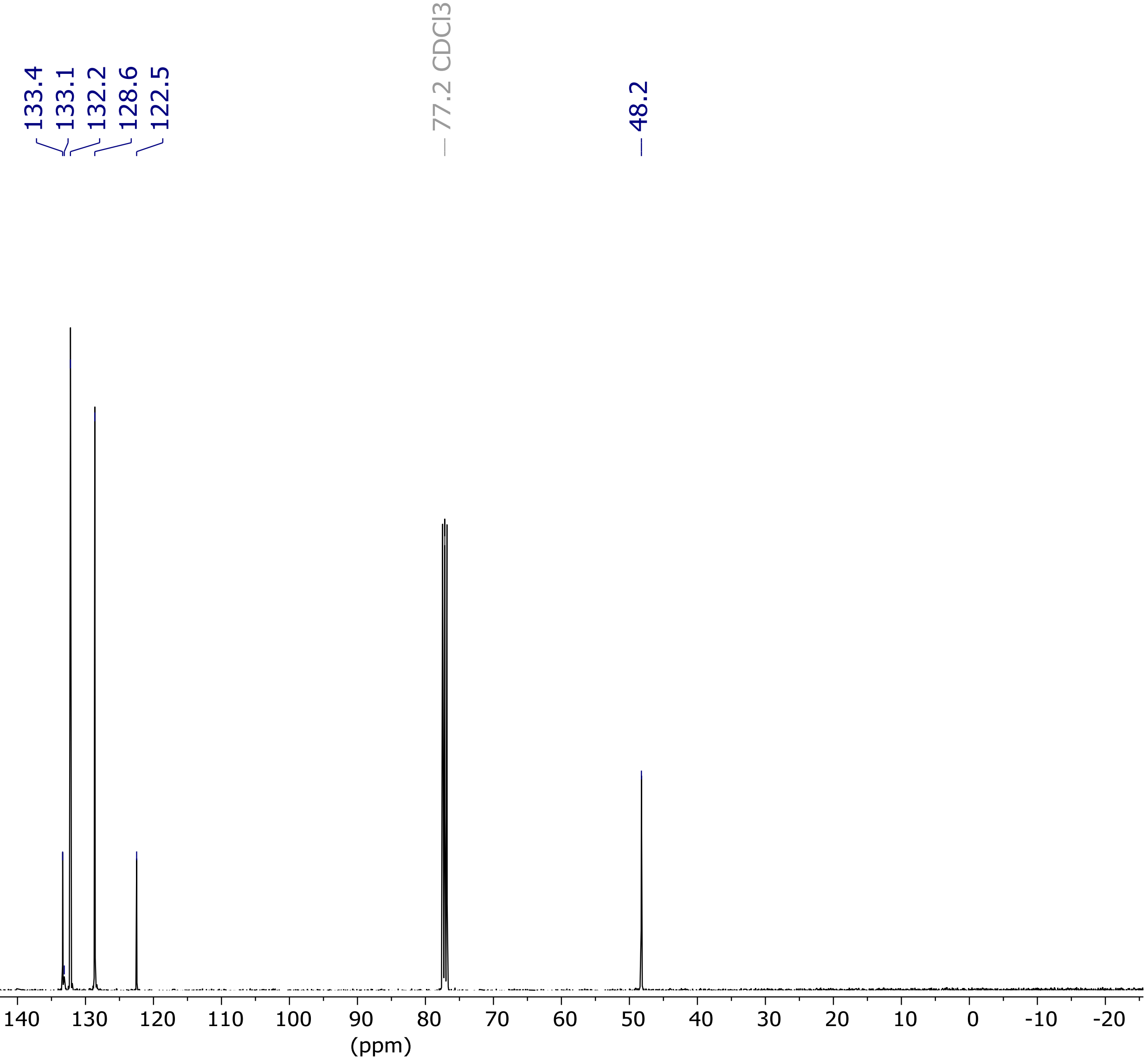
2c

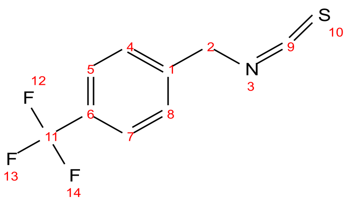




101 MHz, $CDCl_3$

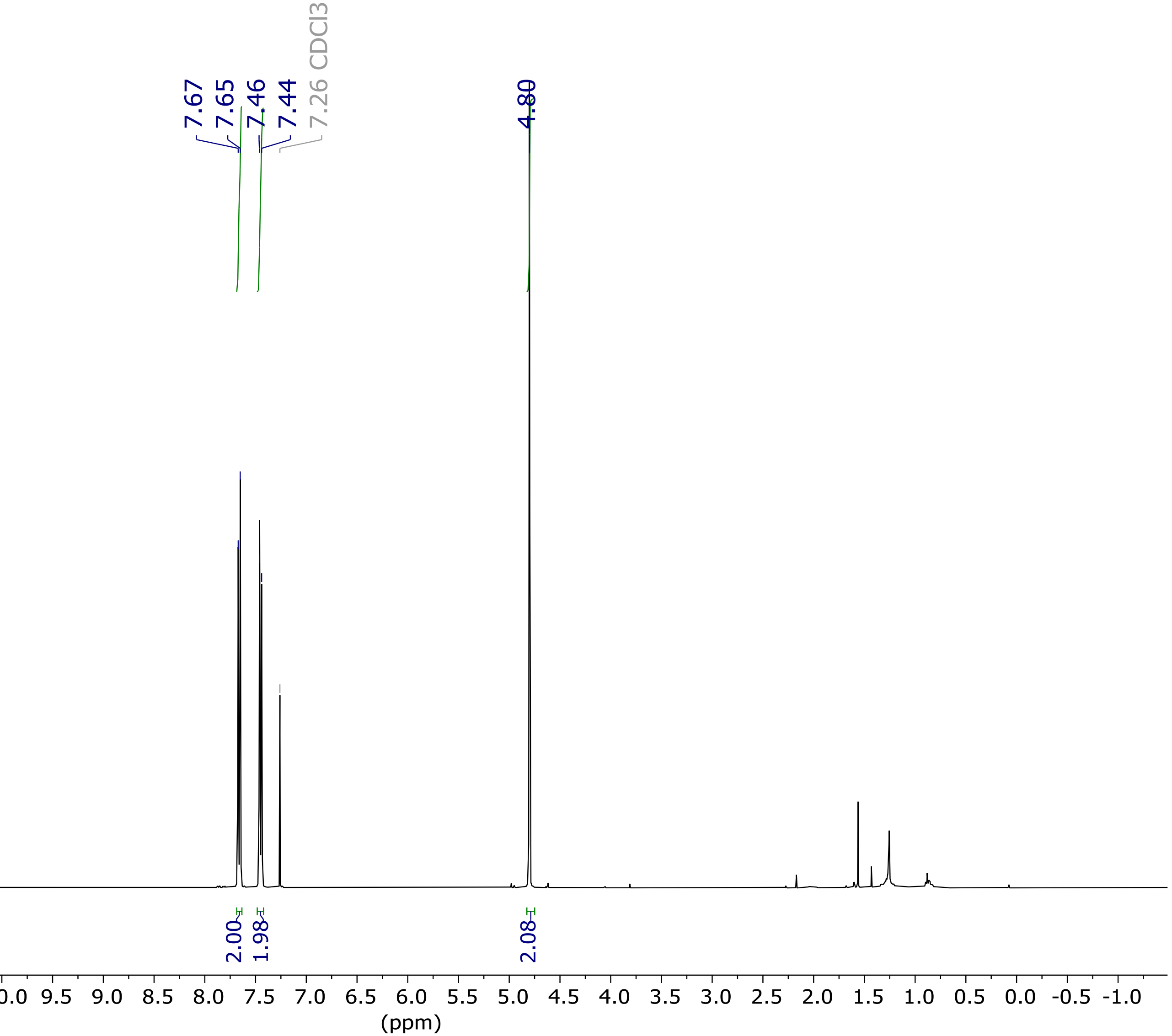
2c

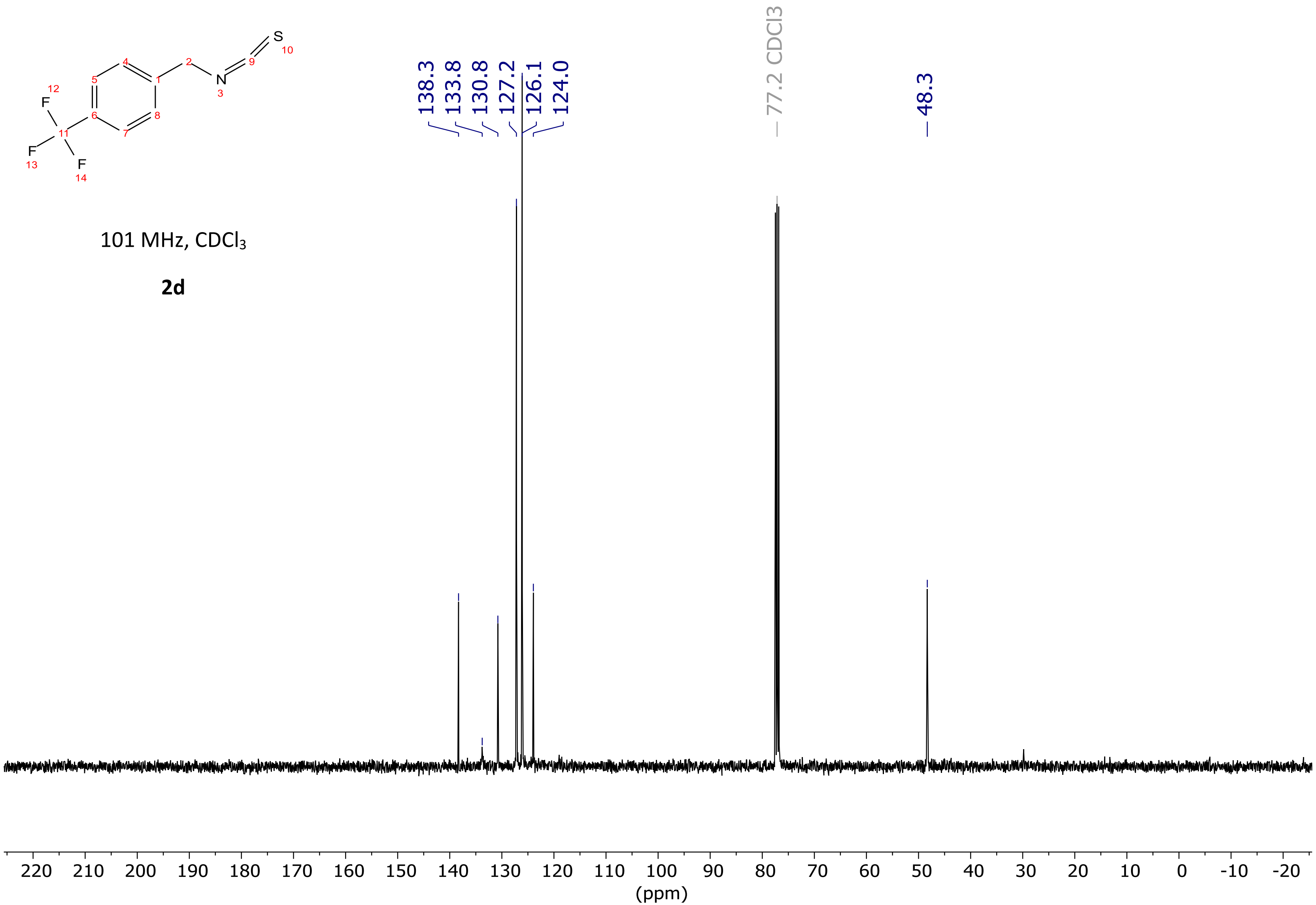




400 MHz, $CDCl_3$

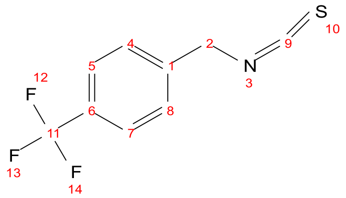
2d





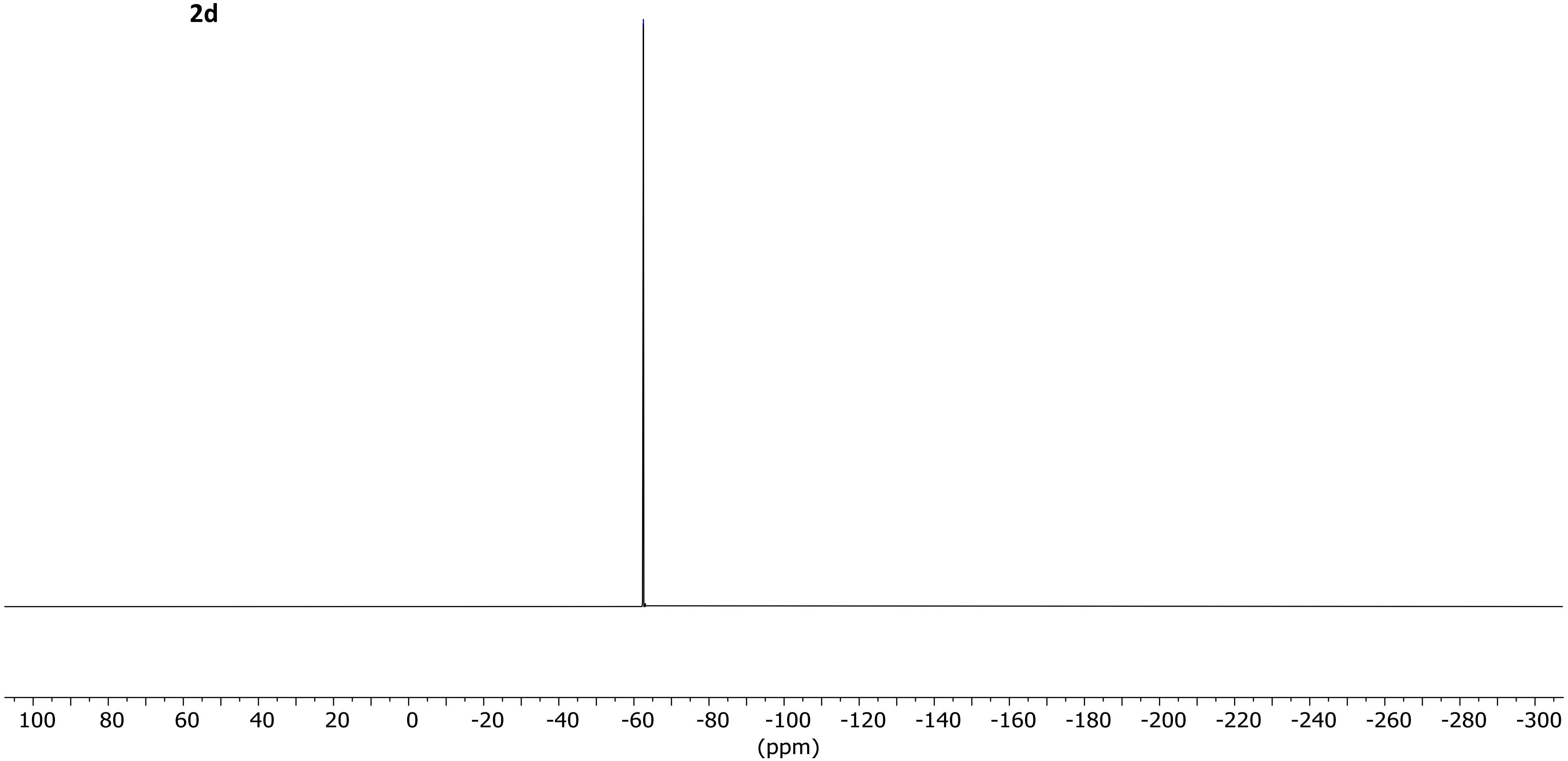
101 MHz, $CDCl_3$

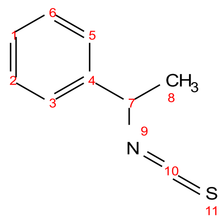
2d



376 MHz, $CDCl_3$

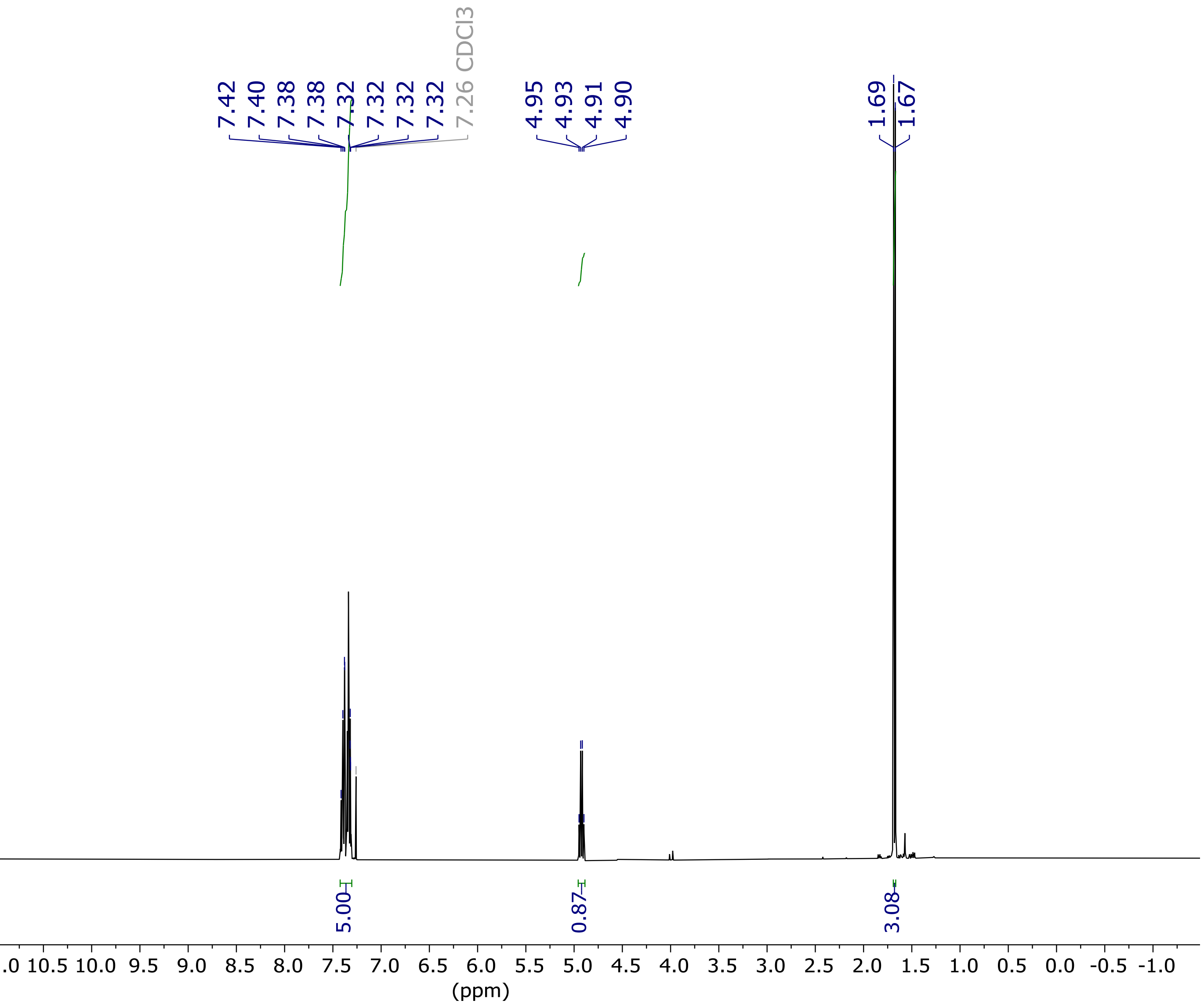
2d

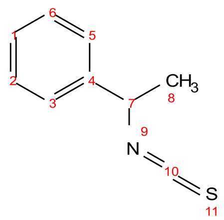




400 MHz, $CDCl_3$

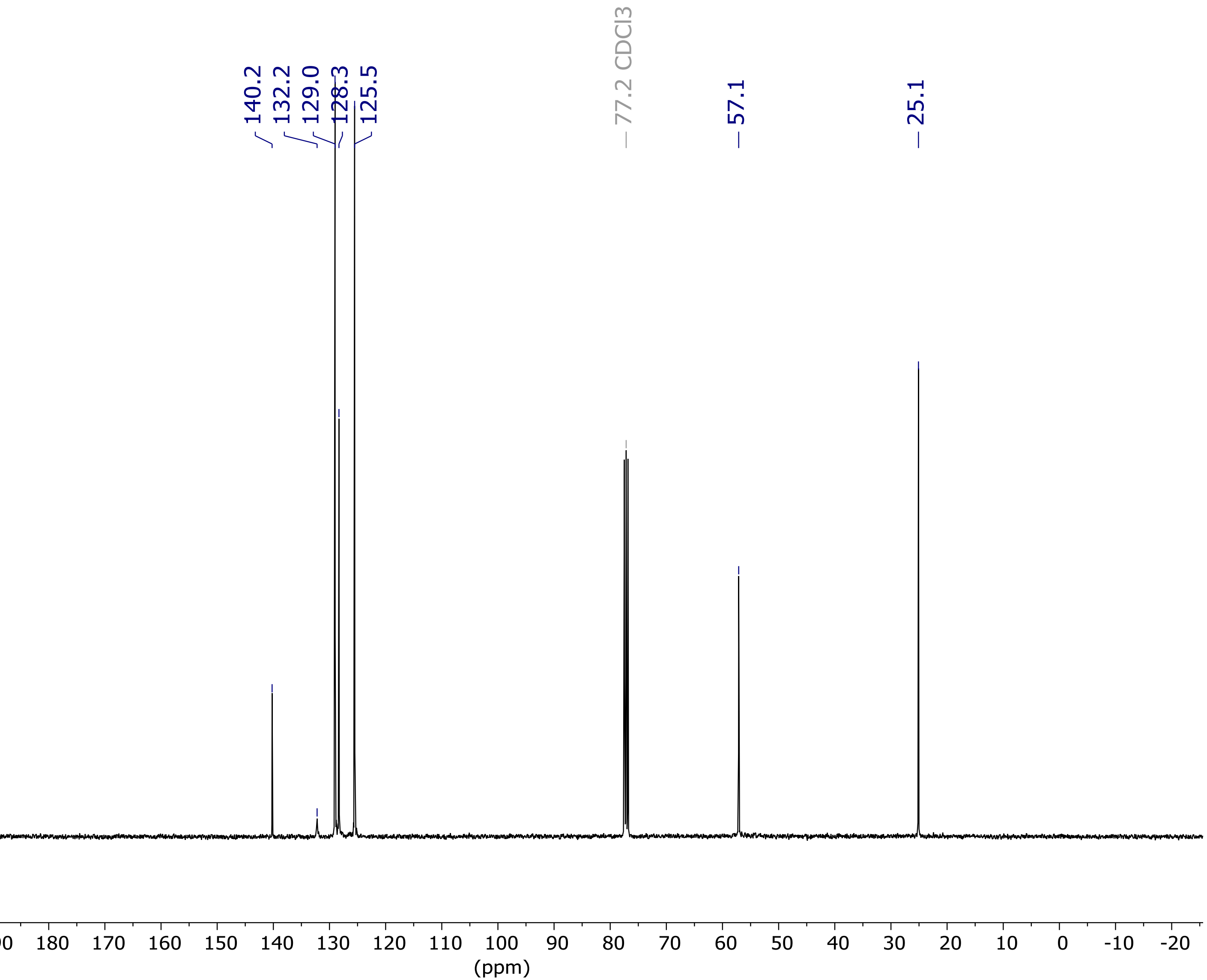
2e

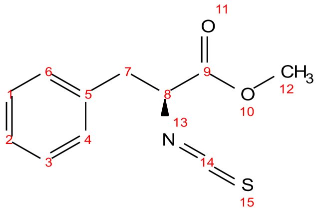




101 MHz, $CDCl_3$

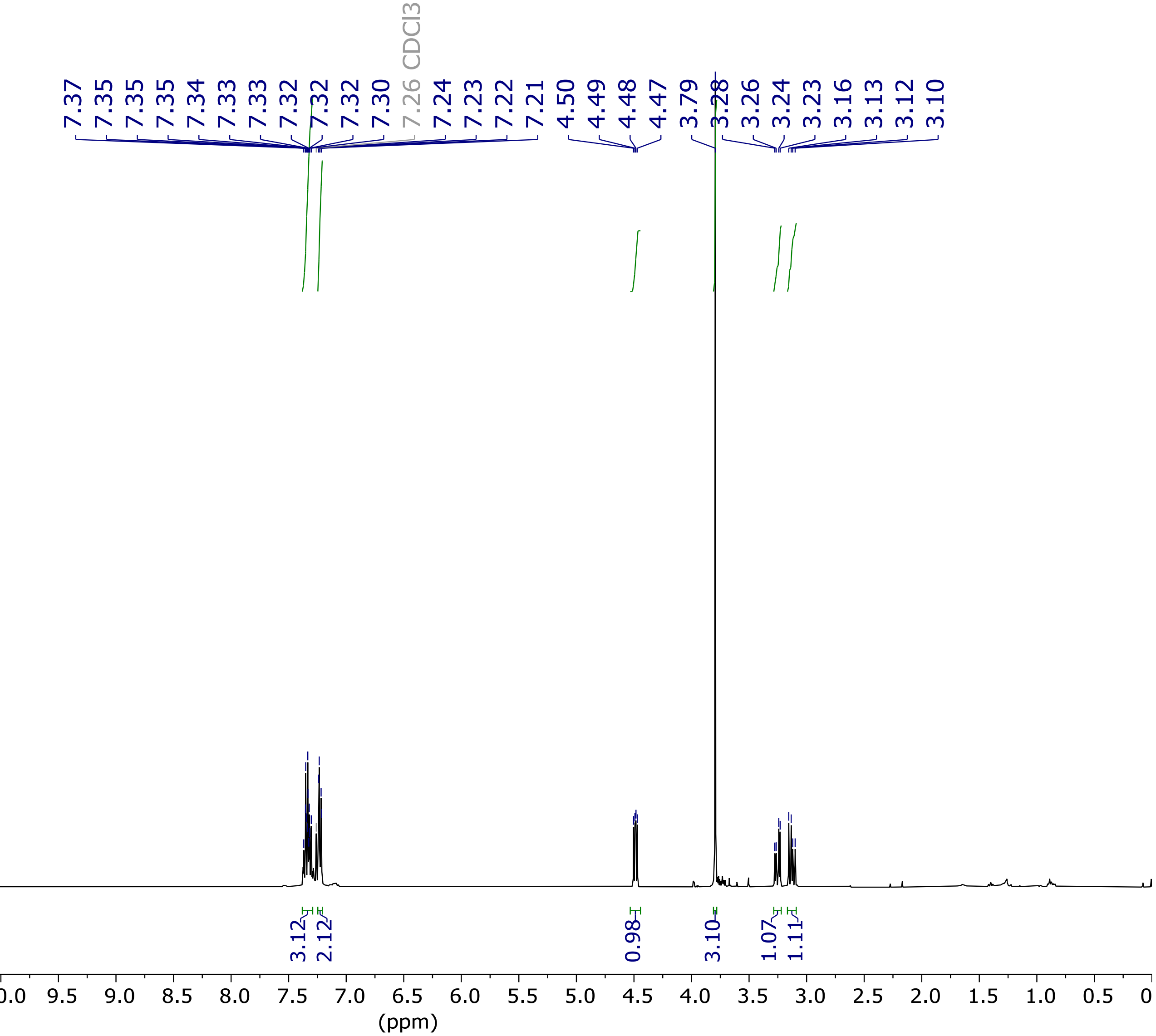
2e

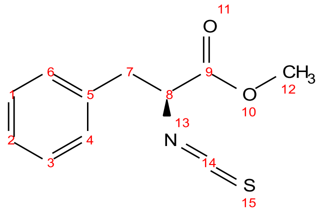




400 MHz, $CDCl_3$

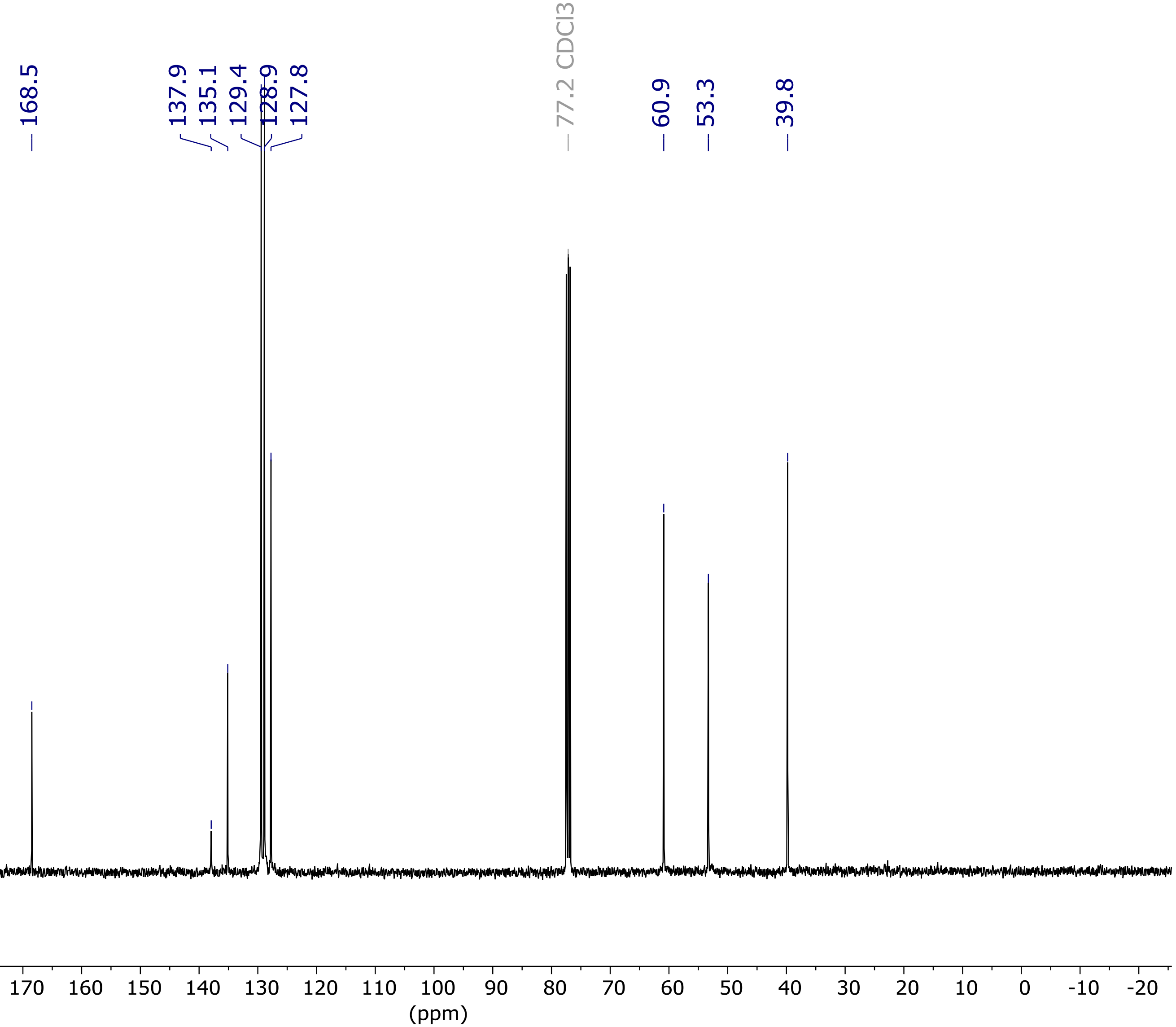
2f

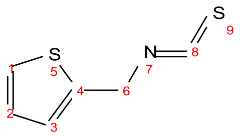




101 MHz, $CDCl_3$

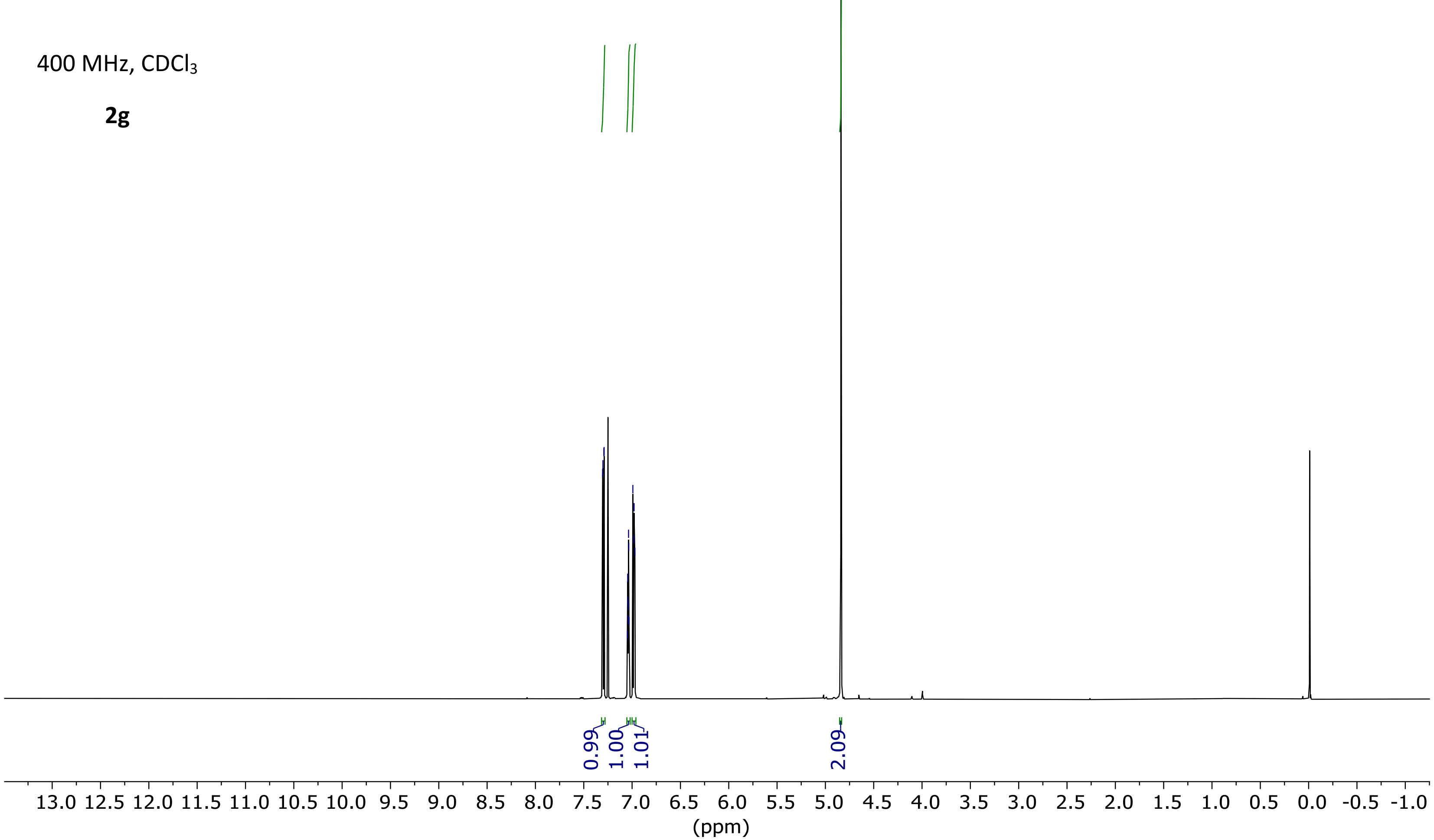
2f

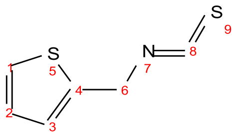




400 MHz, $CDCl_3$

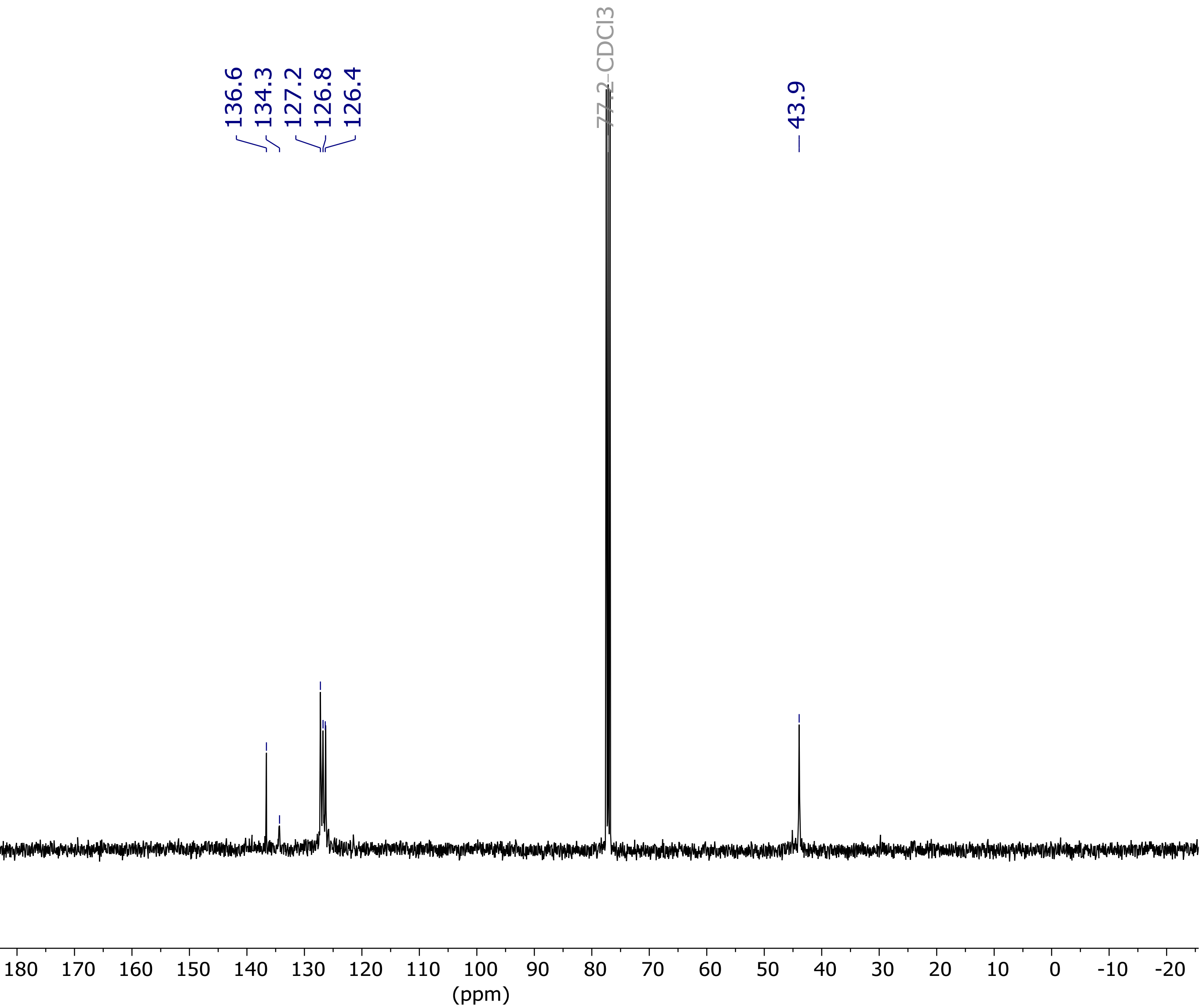
2g

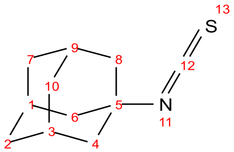




101 MHz, $CDCl_3$

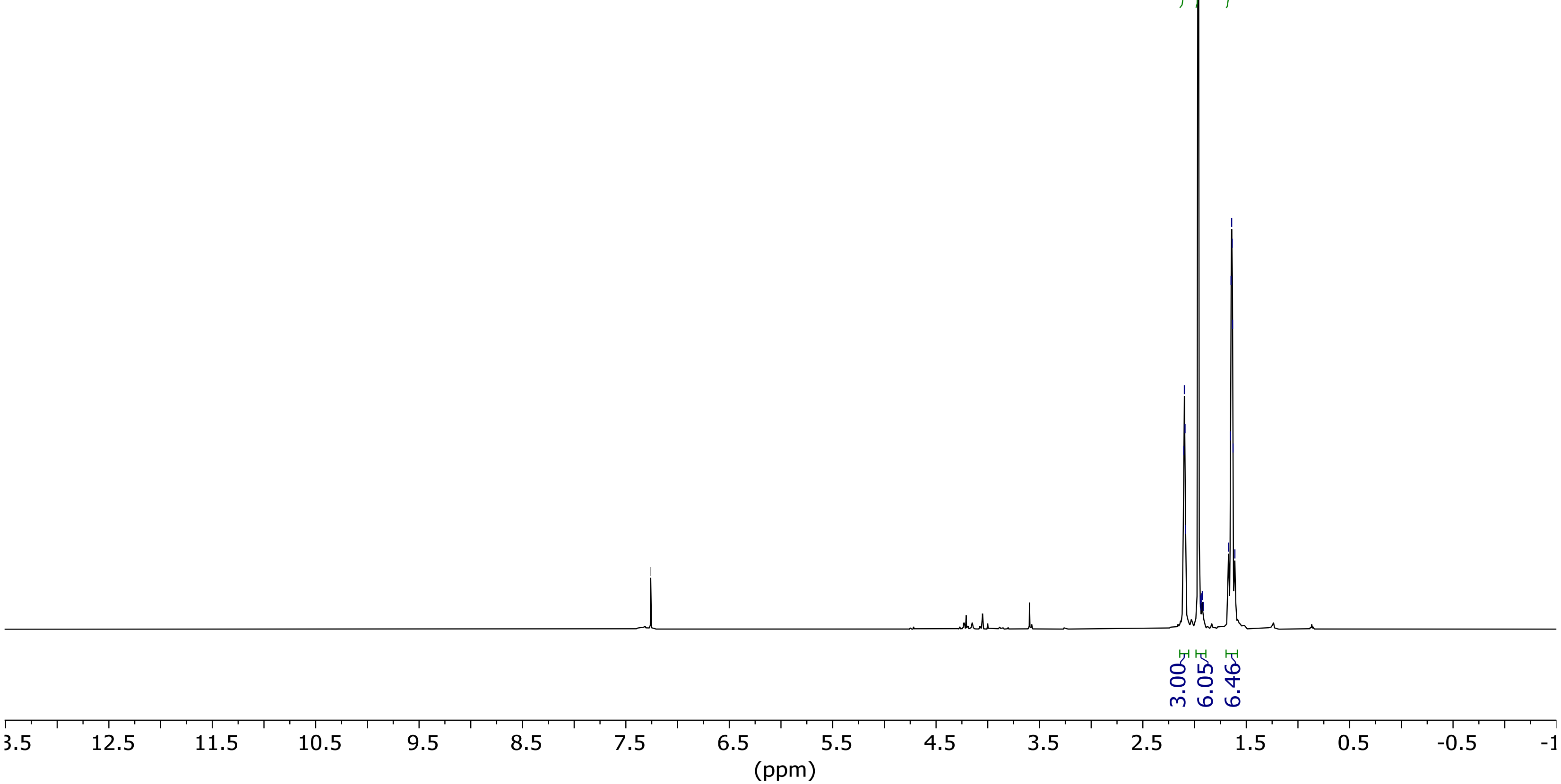
2g

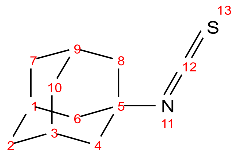




400 MHz, $CDCl_3$

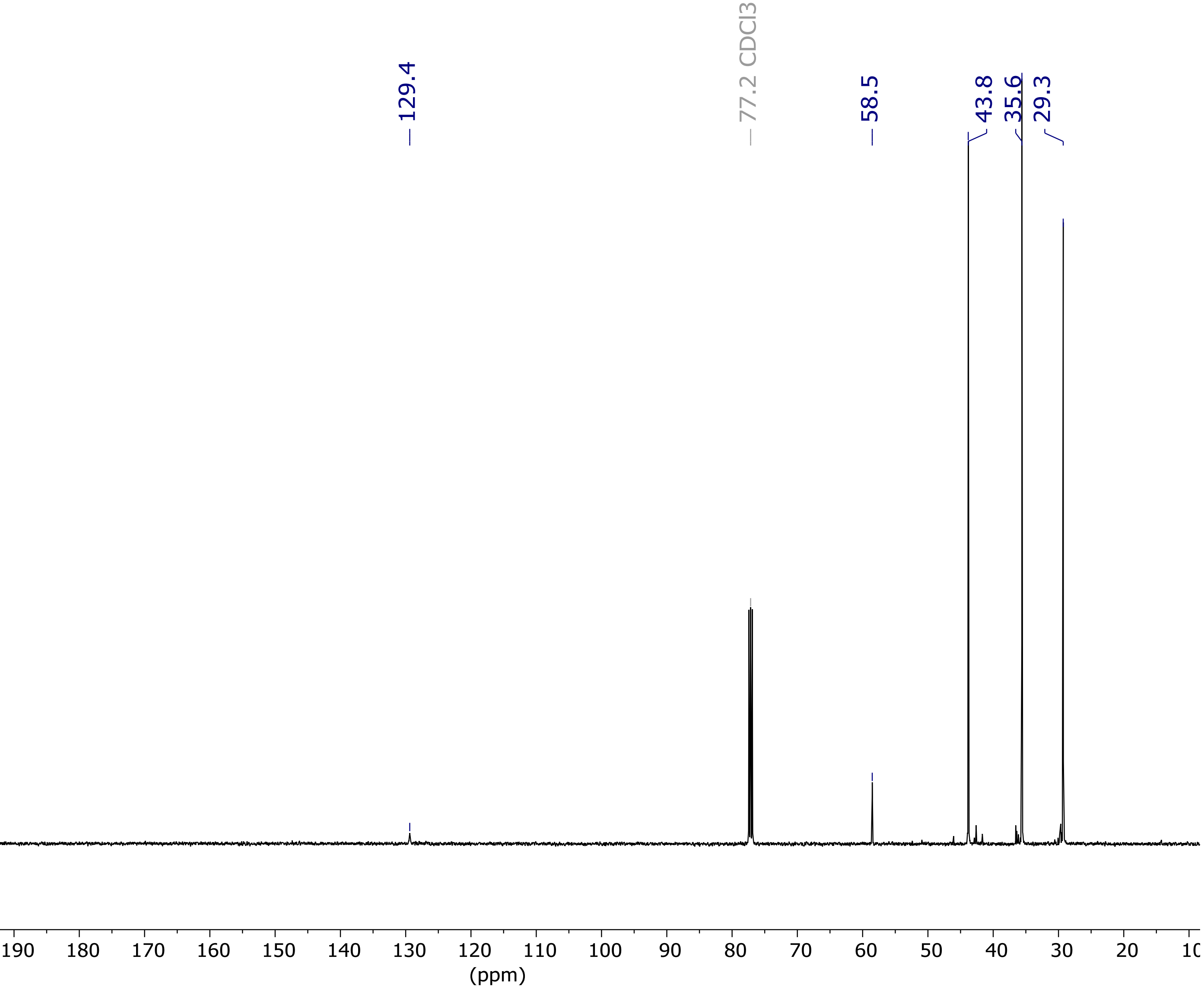
2h

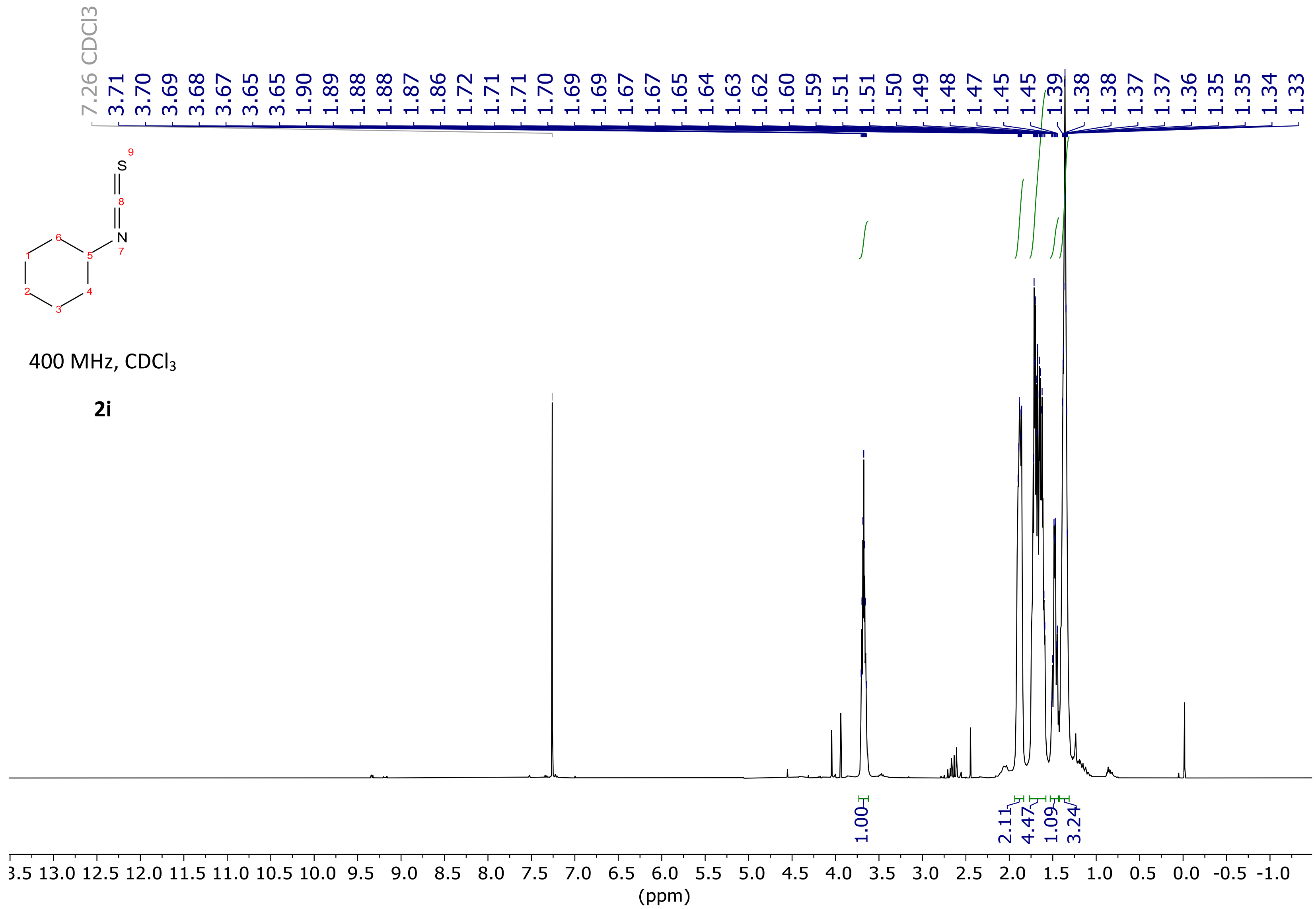




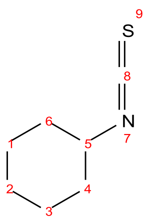
101 MHz, $CDCl_3$

2h



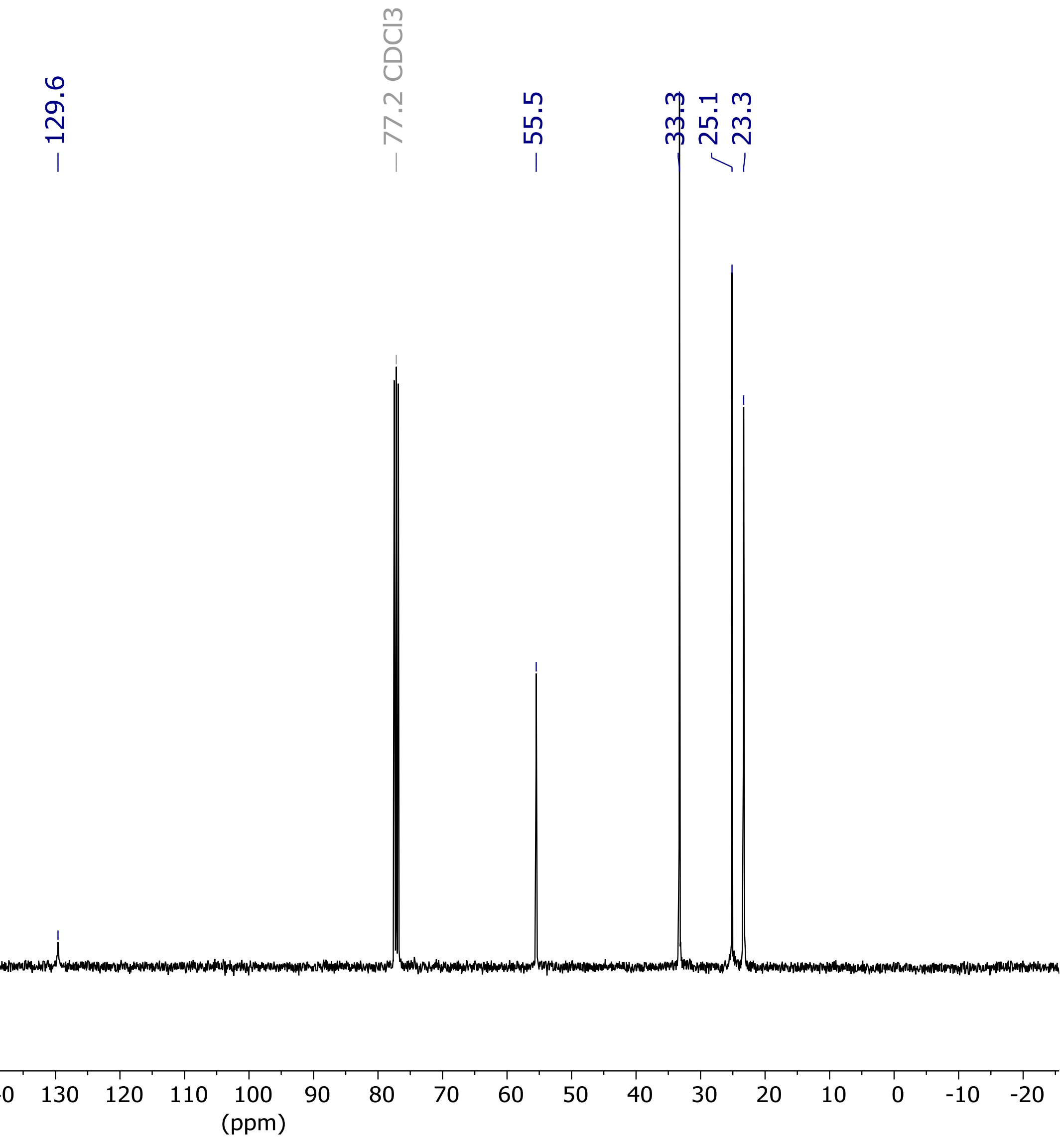


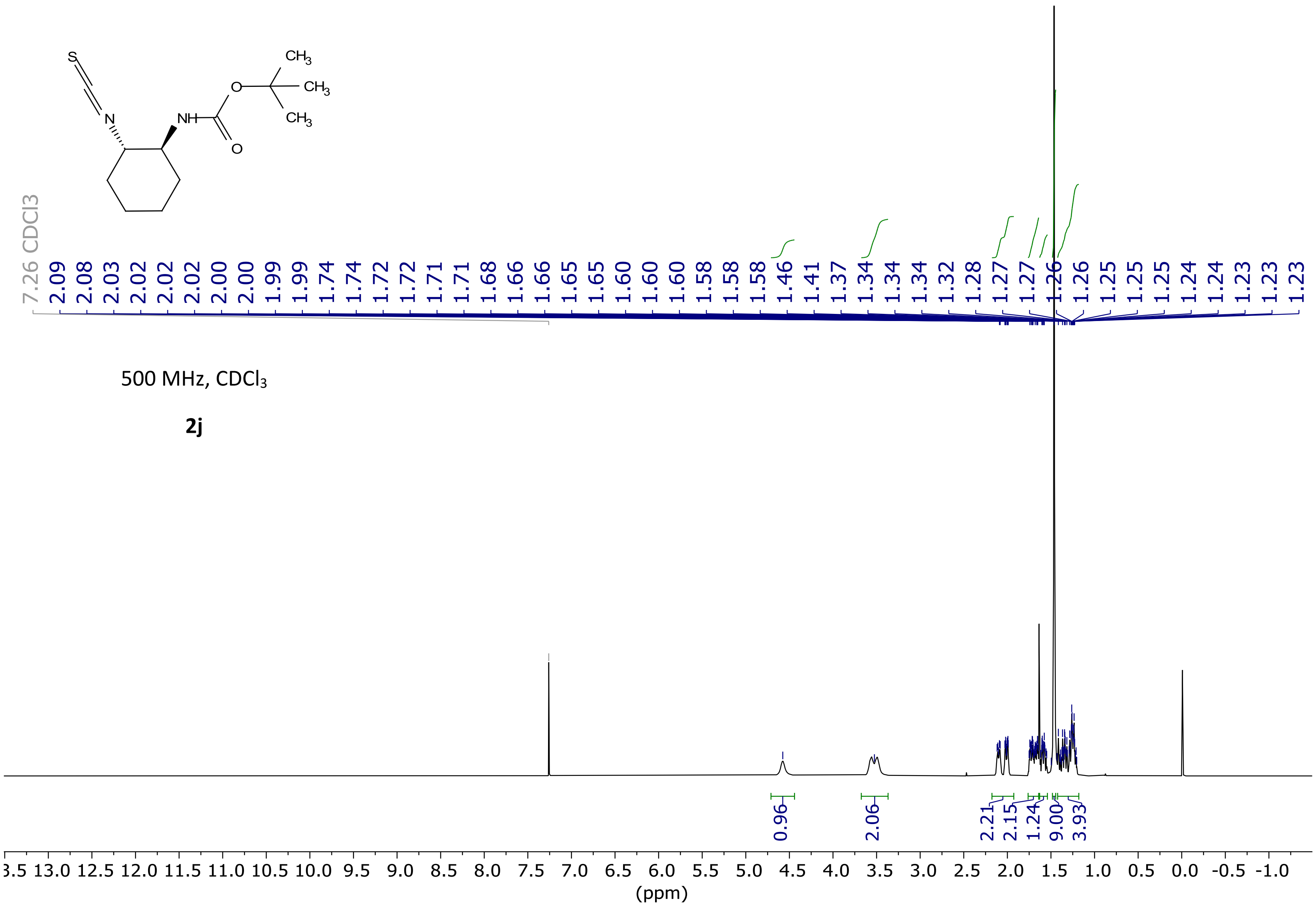
7.26 CDCl3
3.71
3.70
3.69
3.68
3.67
3.65
3.65
1.90
1.89
1.88
1.88
1.87
1.86
1.72
1.71
1.71
1.70
1.69
1.69
1.67
1.67
1.65
1.64
1.63
1.62
1.60
1.59
1.51
1.51
1.50
1.49
1.48
1.47
1.45
1.45
1.39
1.38
1.38
1.37
1.37
1.36
1.35
1.35
1.34
1.33
S
N
400 MHz, CDCl3
2i
1.00
2.11
4.47
1.09
3.24
(ppm)

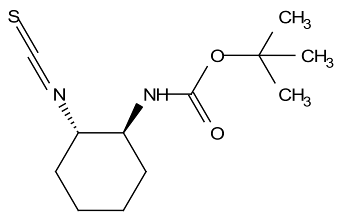


101 MHz, $CDCl_3$

2i

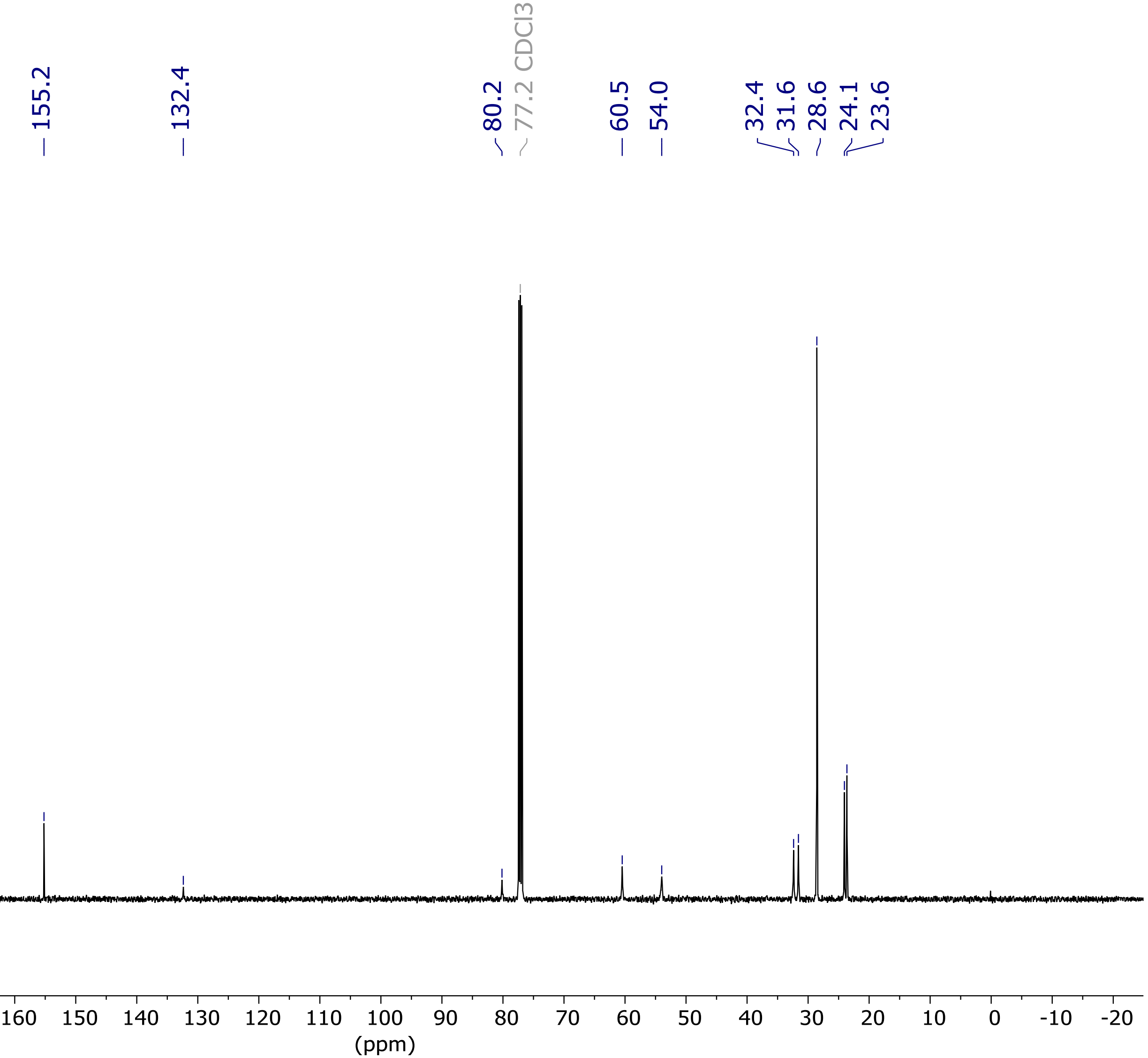


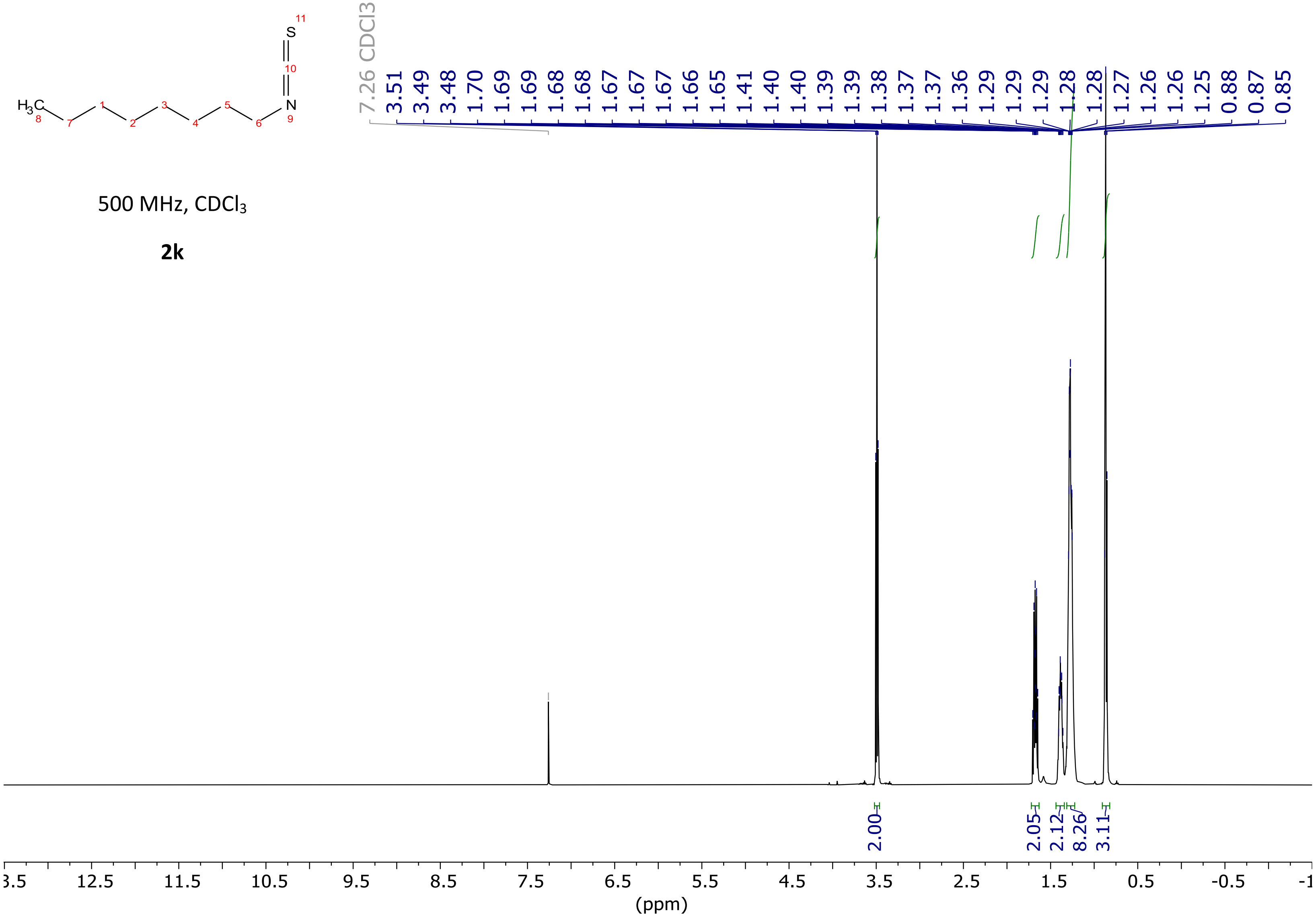

7.26 CDCl3
2.09 2.08 2.03 2.02 2.02 2.02 2.00 2.00 1.99 1.99 1.74 1.74 1.72 1.72 1.71 1.71 1.68 1.66 1.66 1.65 1.65 1.60 1.60 1.60 1.58 1.58 1.58 1.46 1.41 1.37 1.34 1.34 1.34 1.32 1.28 1.27 1.27 1.26 1.26 1.25 1.25 1.25 1.24 1.24 1.23 1.23 1.23
500 MHz, CDCl$_3$
2j
0.96
2.06
2.21
2.15
1.24
9.00
3.93
(ppm)

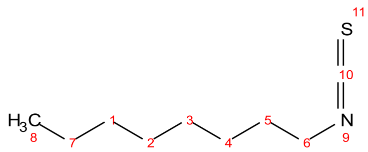


126 MHz, $CDCl_3$

2j

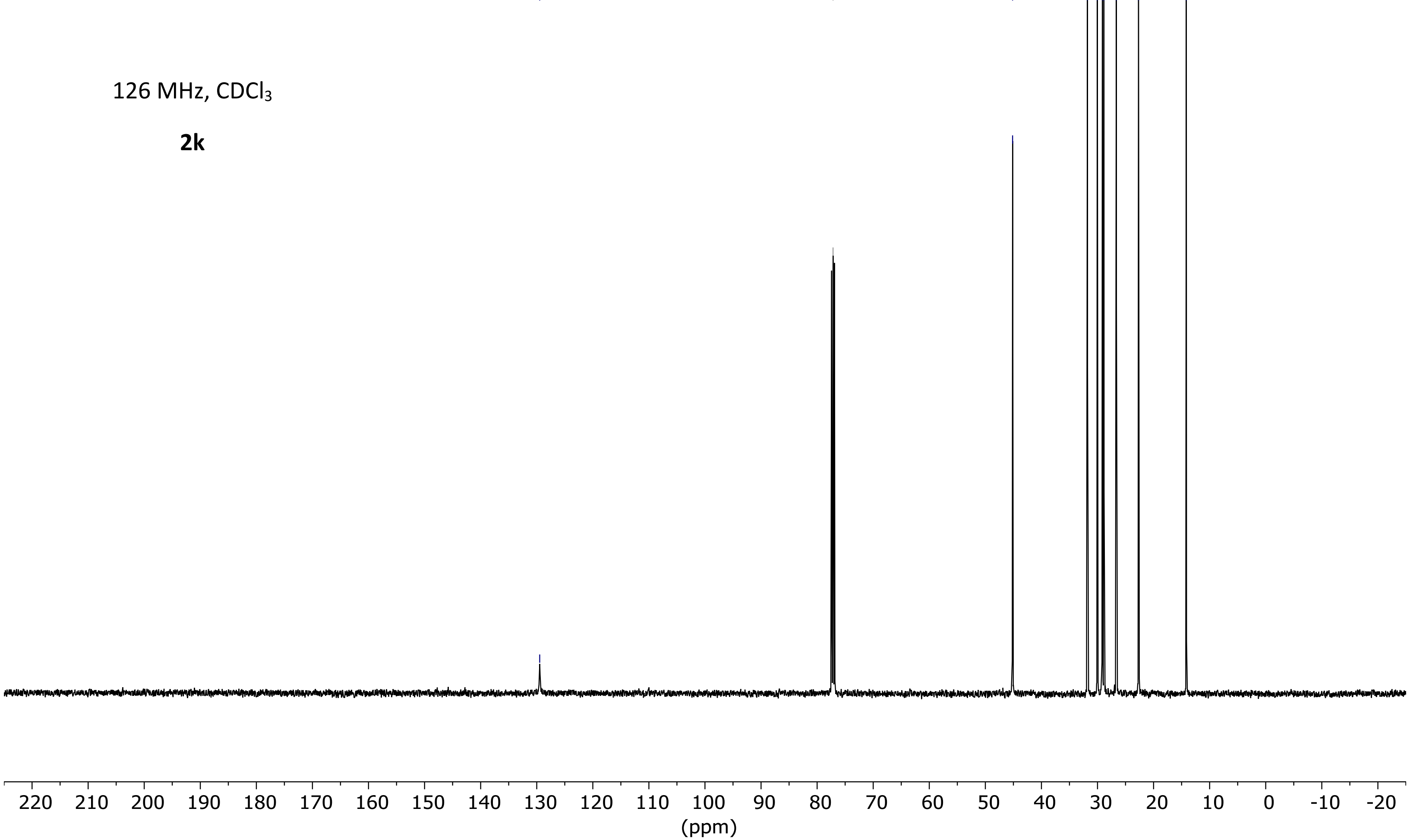


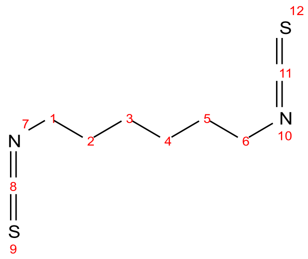

H3C
S
N
500 MHz, CDCl3
2k
7.26 CDCl3
3.51
3.49
3.48
1.70
1.69
1.69
1.68
1.68
1.67
1.67
1.67
1.66
1.65
1.41
1.40
1.40
1.39
1.39
1.38
1.37
1.37
1.36
1.29
1.29
1.29
1.28
1.28
1.27
1.26
1.26
1.25
0.88
0.87
0.85
2.00
2.05
2.12
8.26
3.11
(ppm)



126 MHz, $CDCl_3$

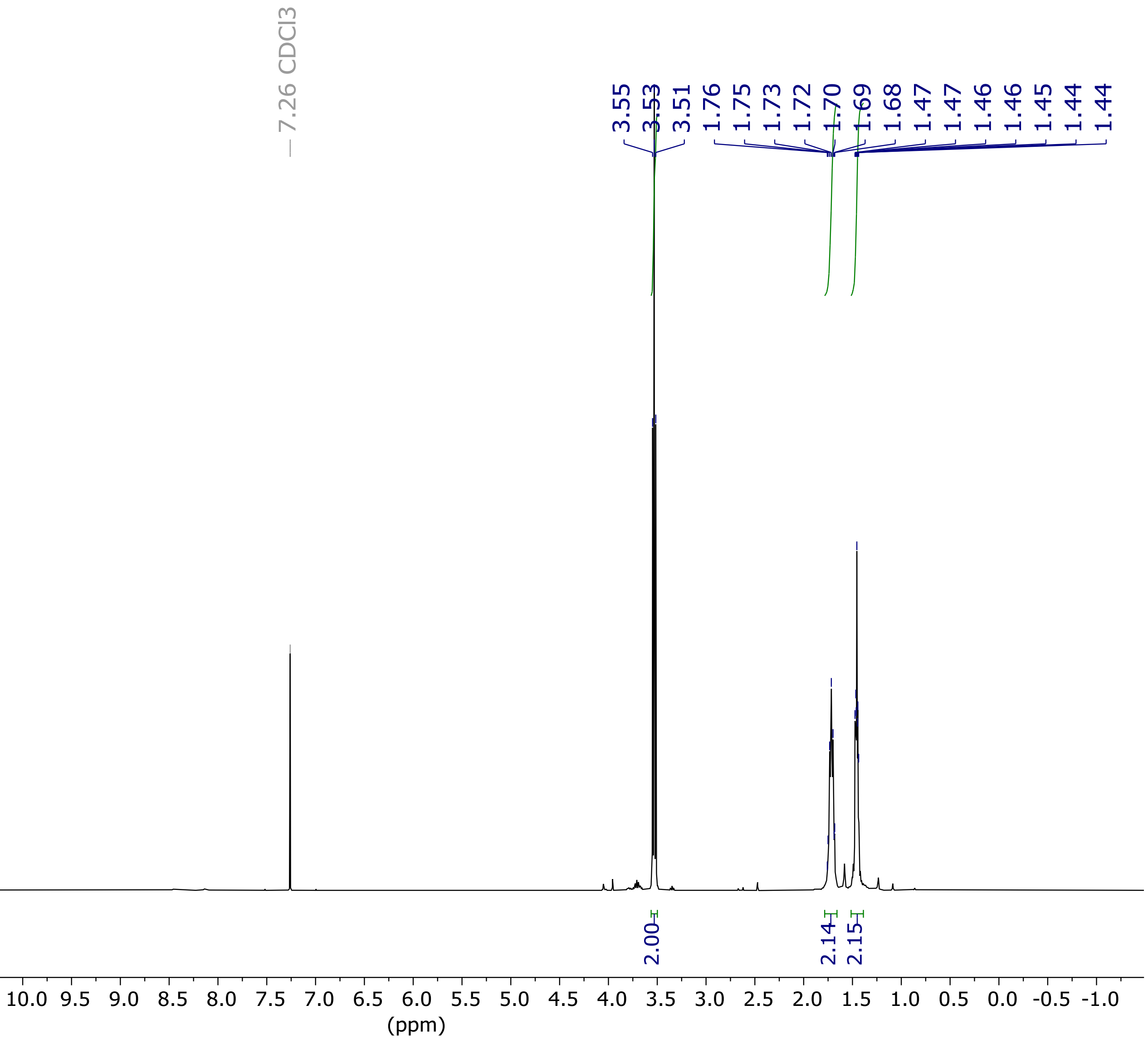
2k

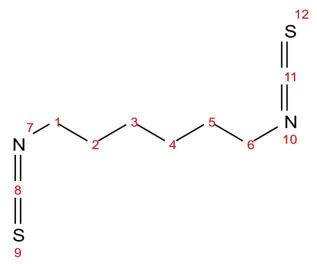




500 MHz, $CDCl_3$

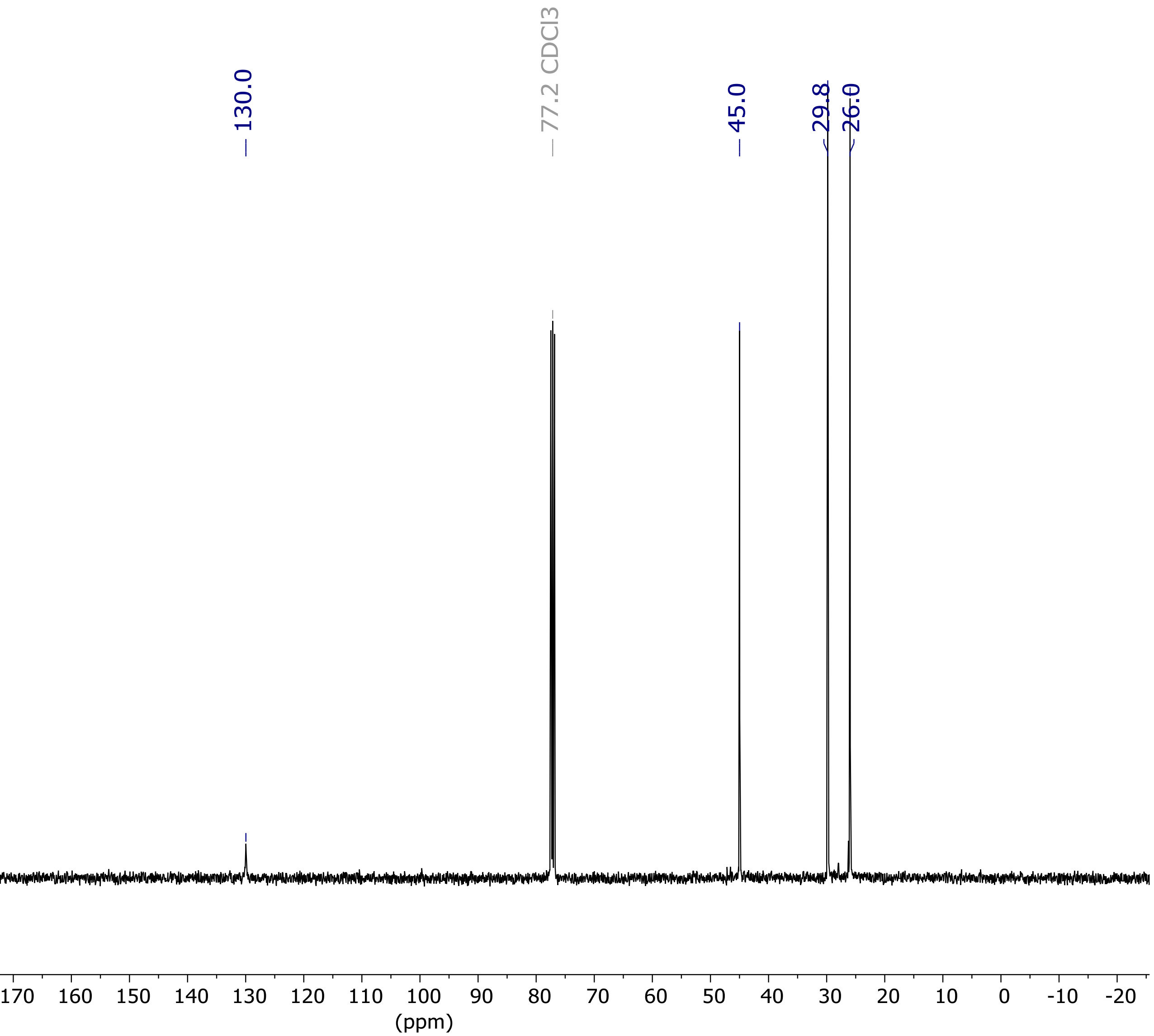
2l

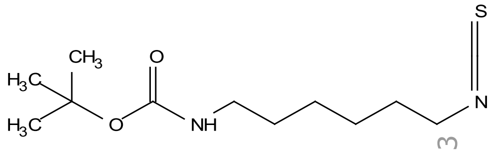




126 MHz, $CDCl_3$

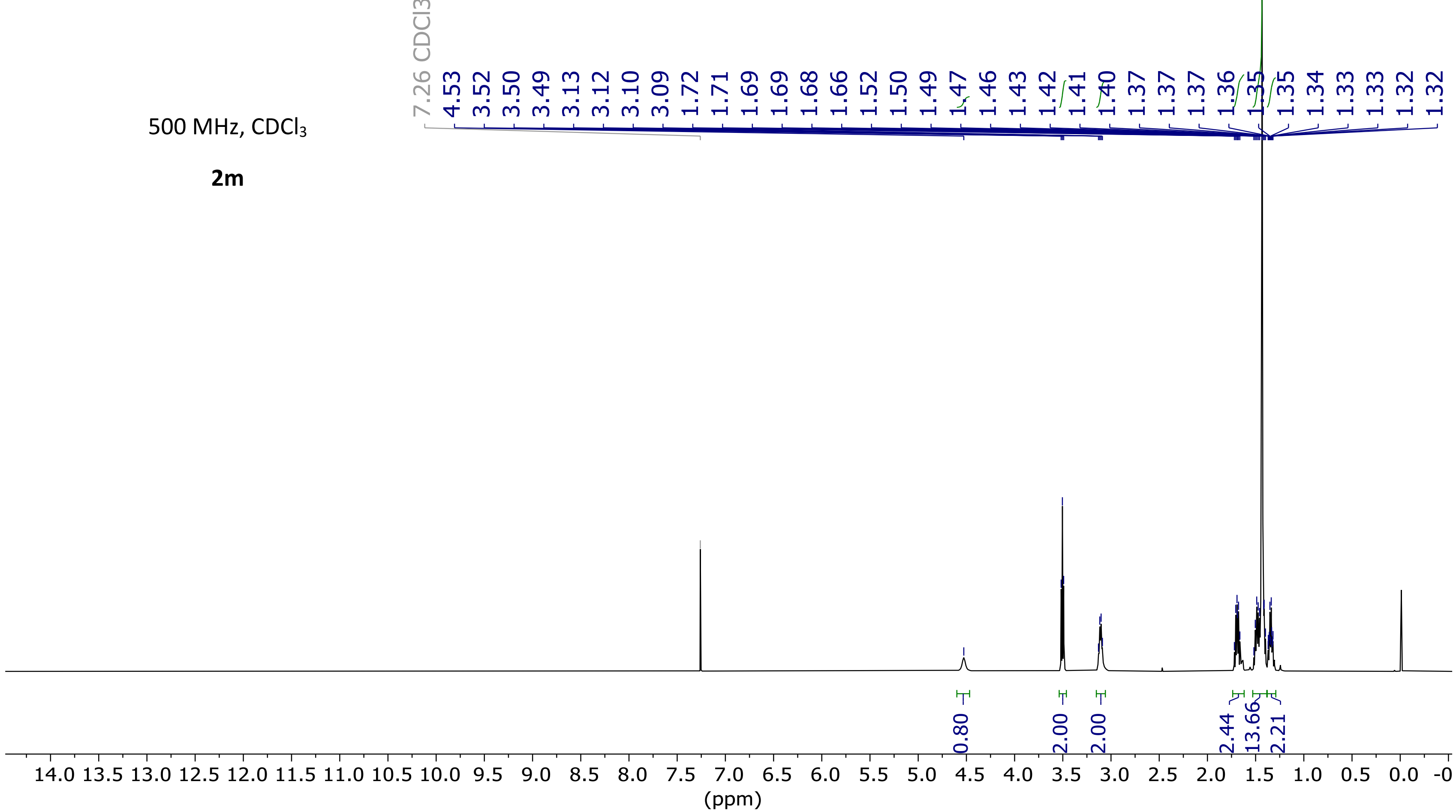
2l

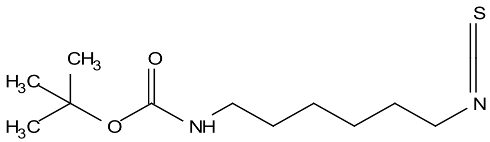




500 MHz, $CDCl_3$

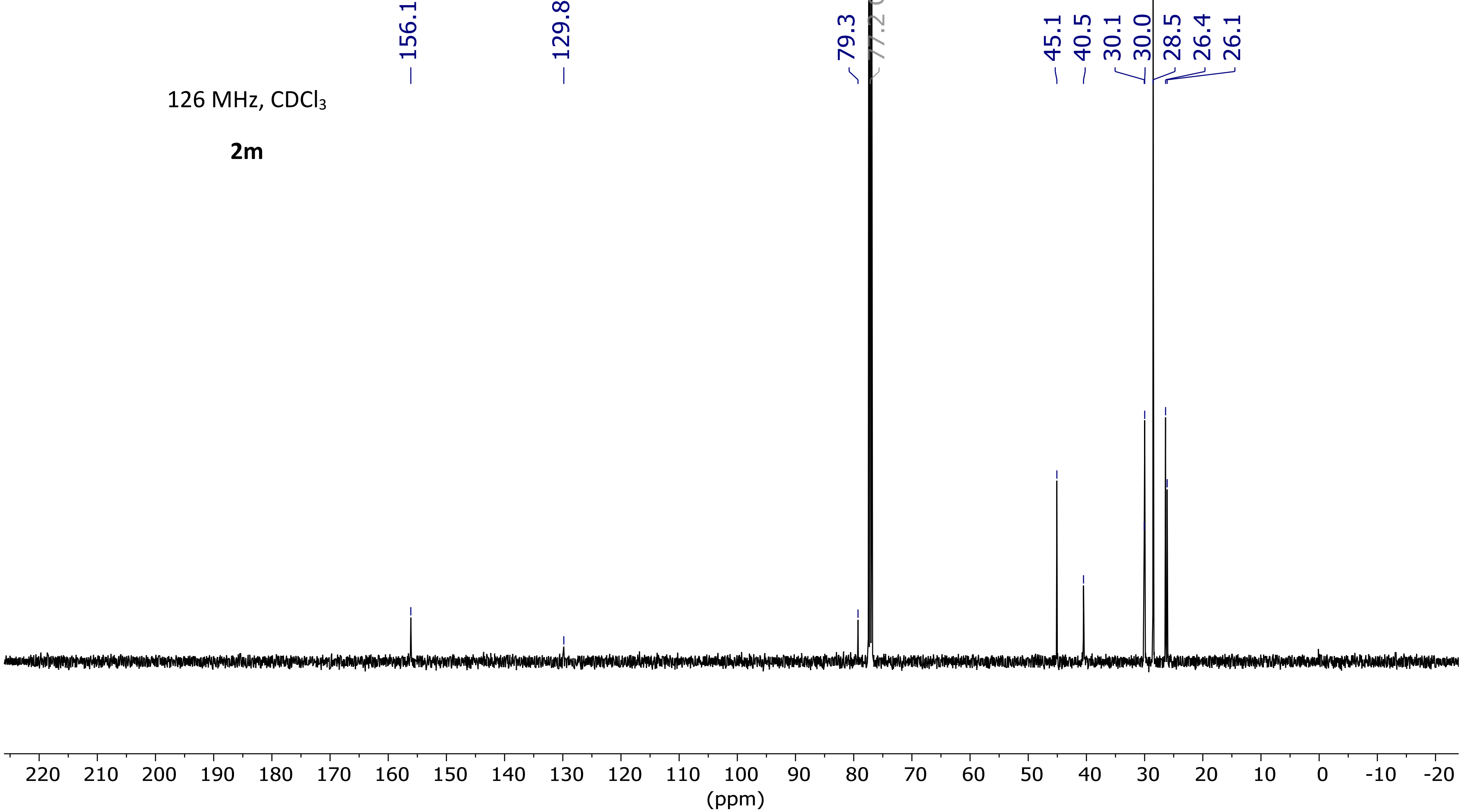
2m

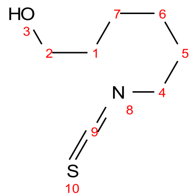




126 MHz, $CDCl_3$

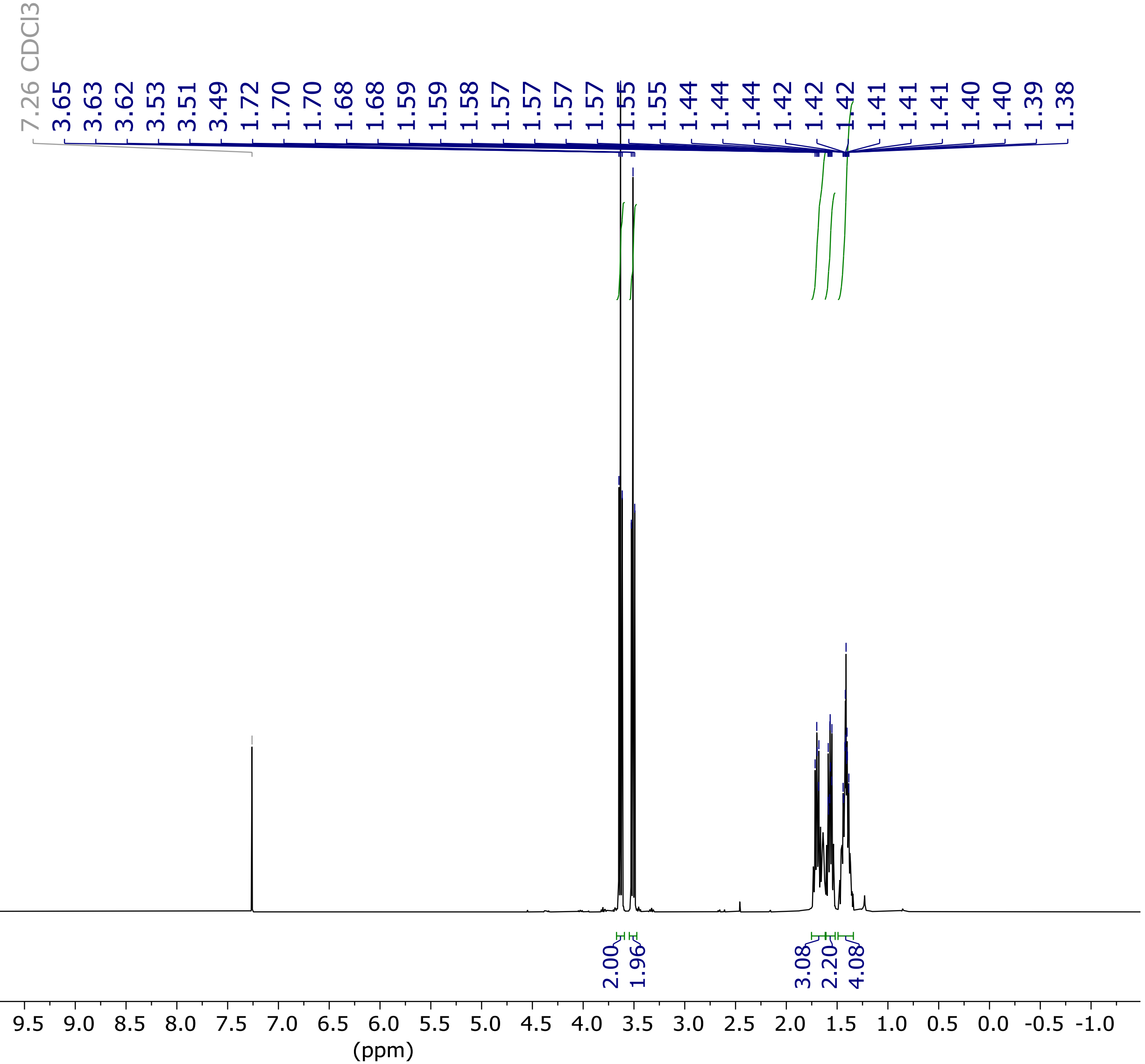
2m

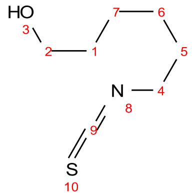




500 MHz, $CDCl_3$

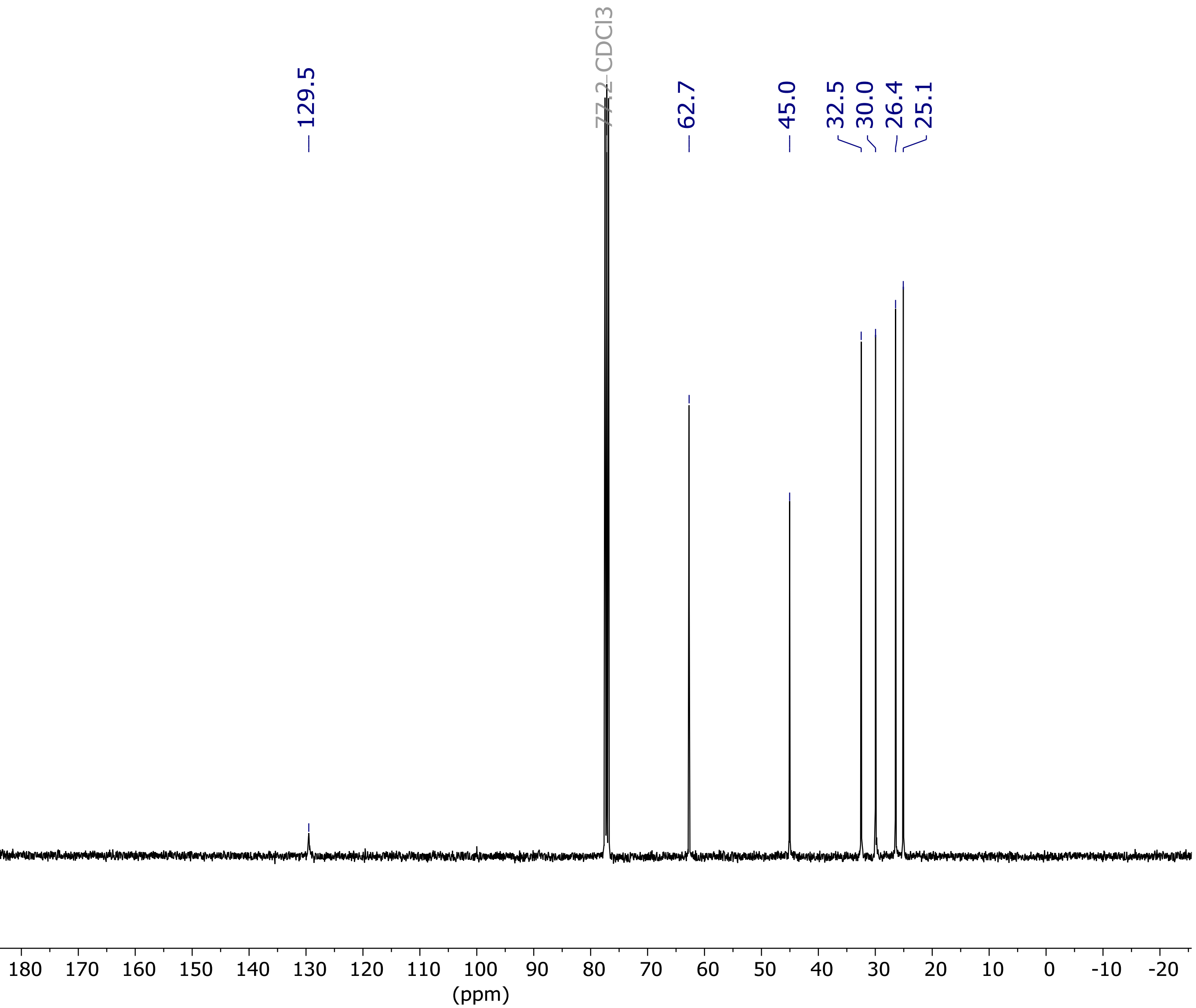
2n

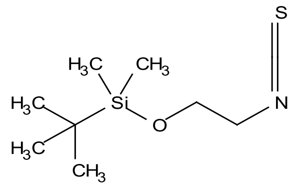




126 MHz, $CDCl_3$

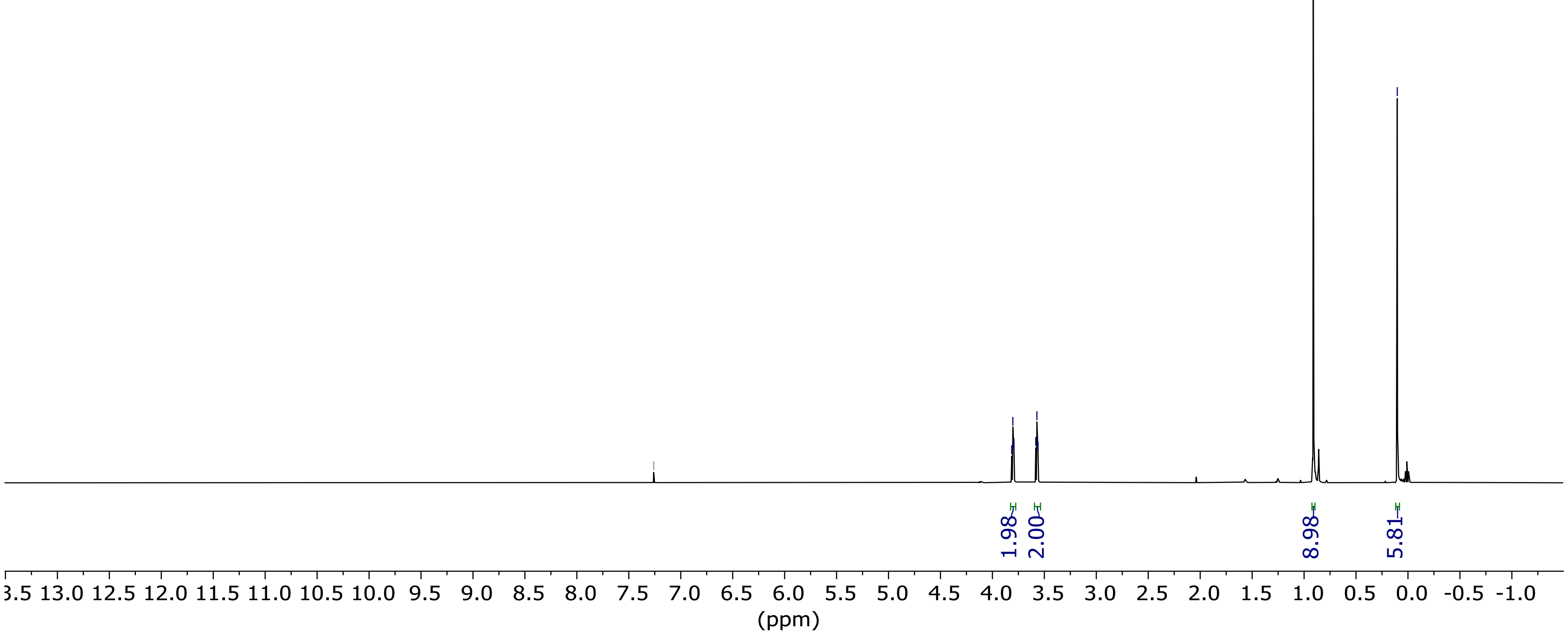
2n

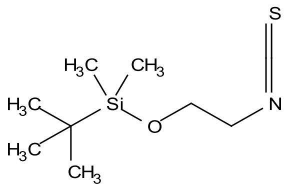




500 MHz, $CDCl_3$

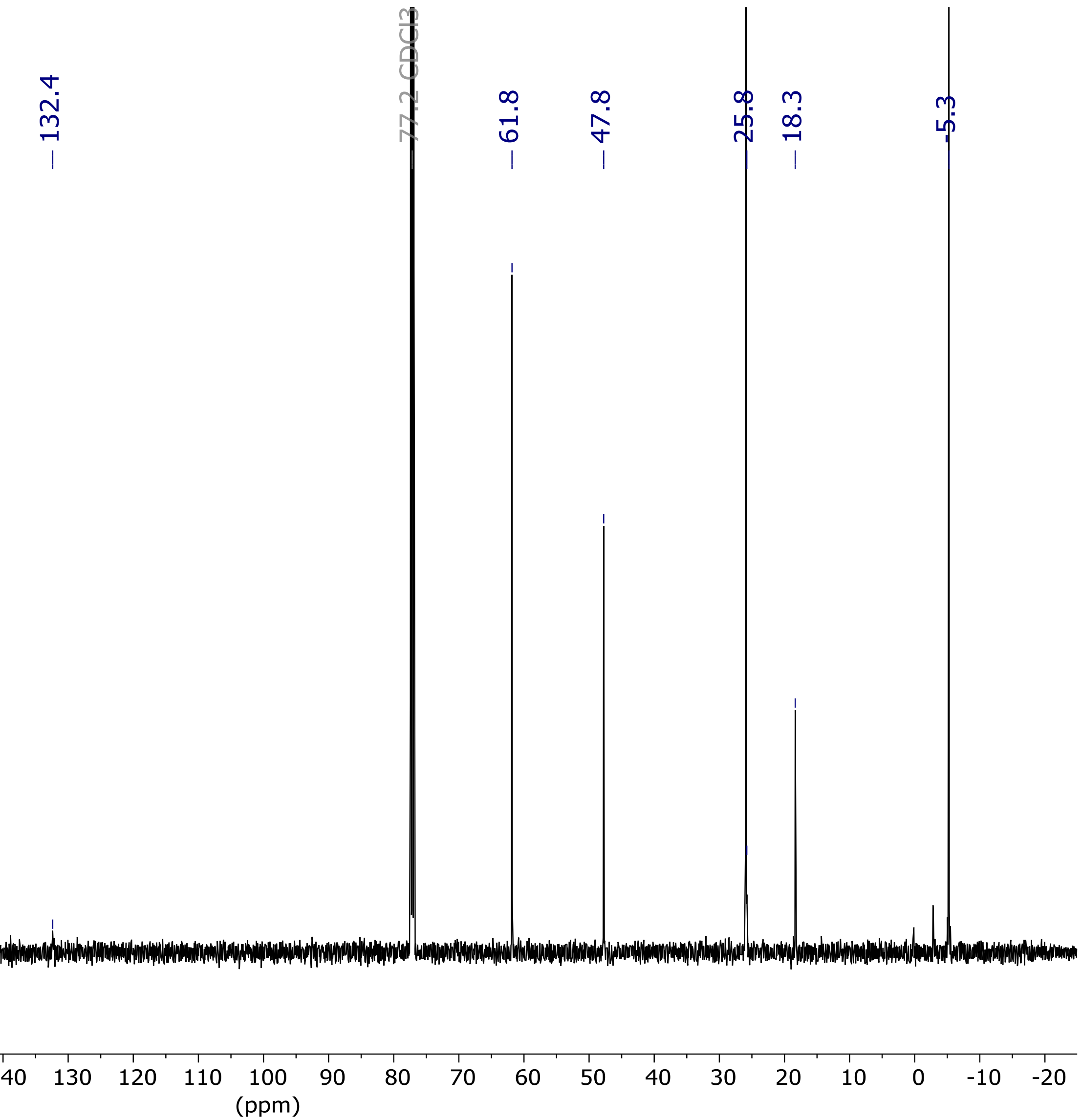
2o

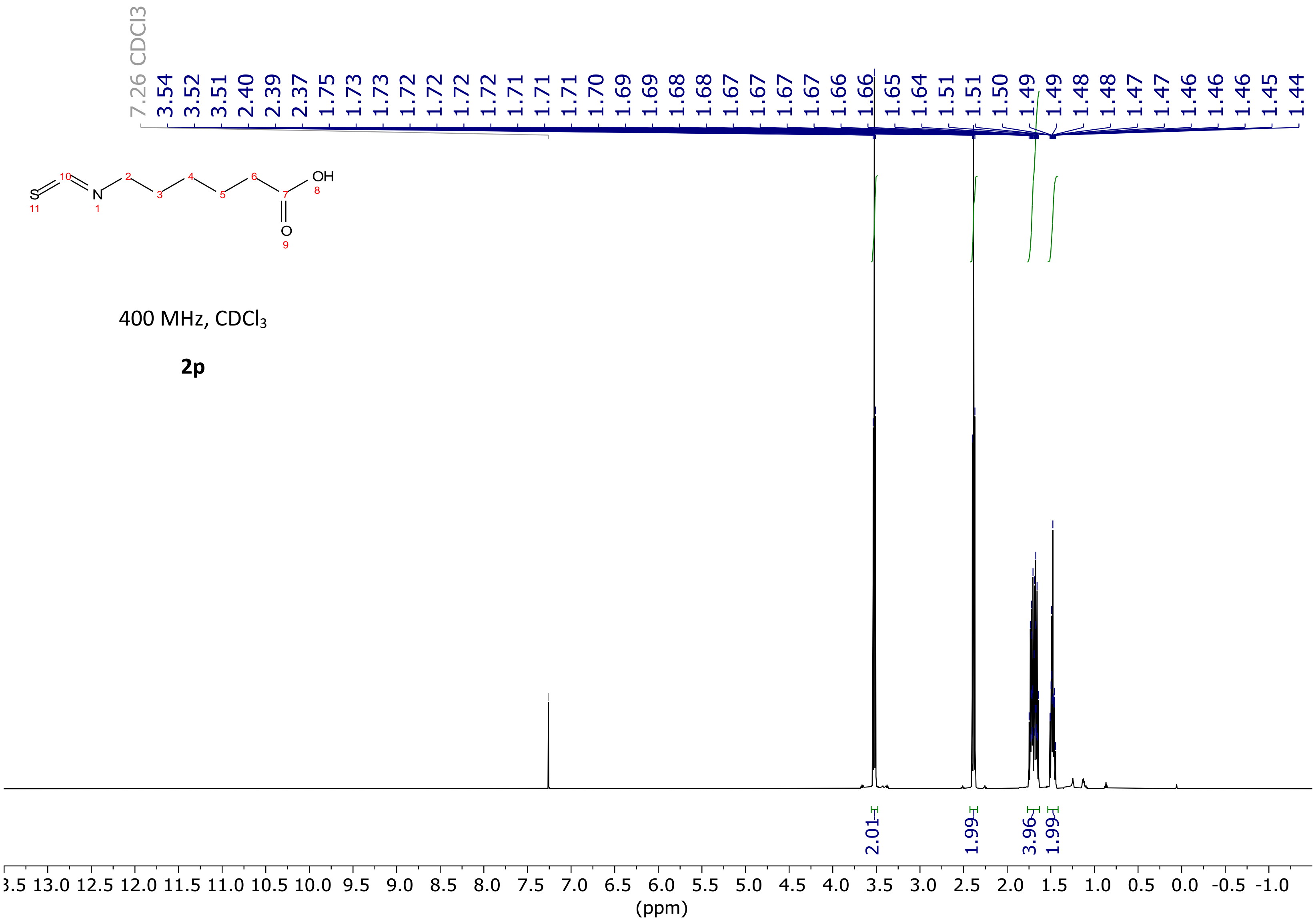




126 MHz, CDCl₃

2o




7.26 CDCl3
3.54
3.52
3.51
2.40
2.39
2.37
1.75
1.73
1.73
1.72
1.72
1.72
1.72
1.71
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1.48
1.48
1.47
1.47
1.46
1.46
1.46
1.45
1.44
S
N
OH
O
400 MHz, $CDCl_3$
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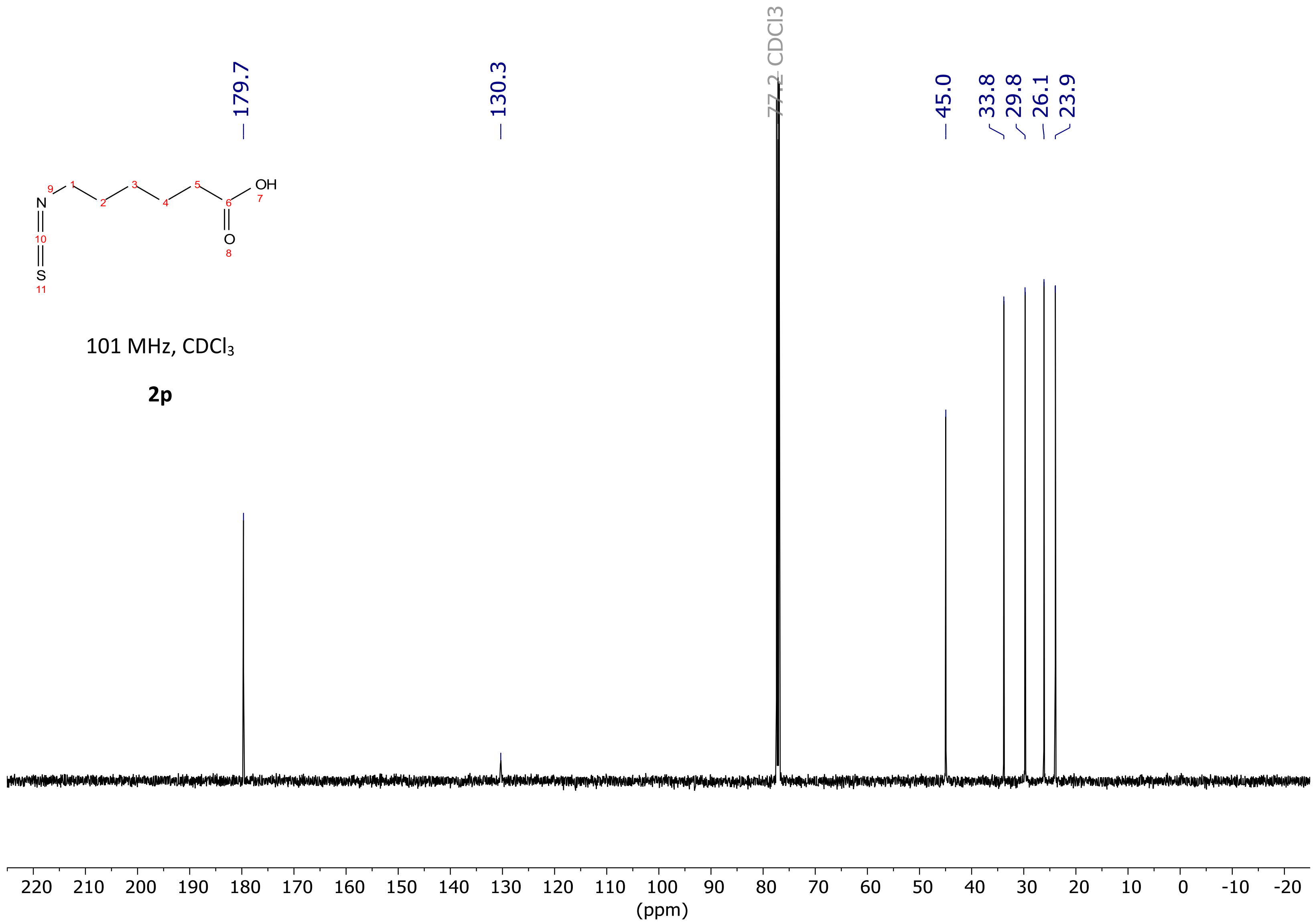
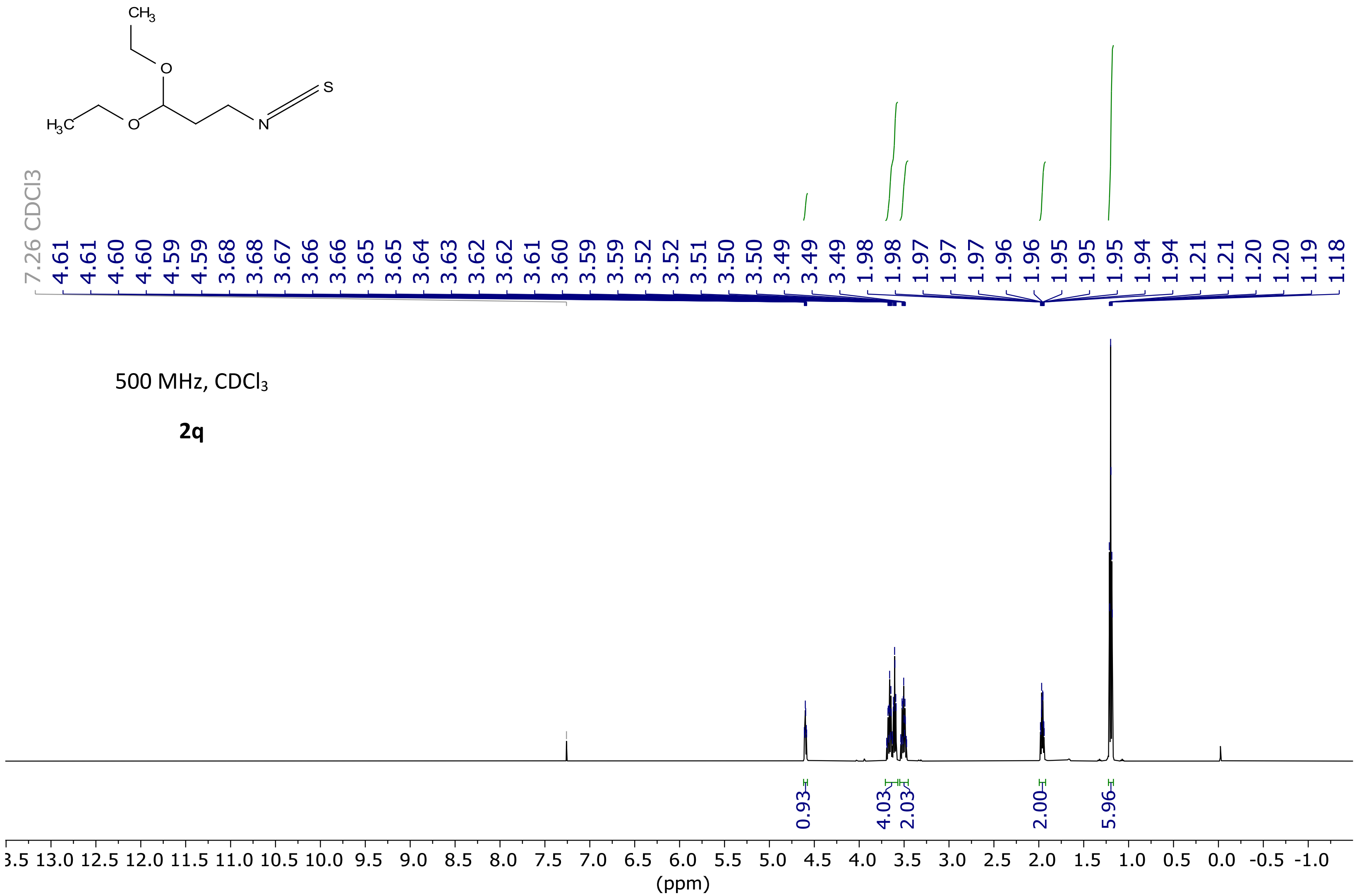
2.01
1.99
3.96
1.99
(ppm)


179.7
130.3
77.2 CDCl3
45.0
33.8
29.8
26.1
23.9
N
S
OH
O
101 MHz, CDCl3
2p
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20
(ppm)



CH3
O
S
H3C
O
N
7.26 CDCl3
4.61
4.61
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3.68
3.67
3.66
3.66
3.65
3.65
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1.97
1.96
1.96
1.95
1.95
1.95
1.94
1.94
1.21
1.21
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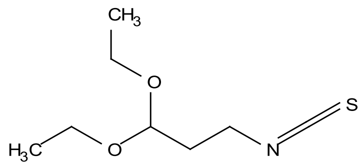
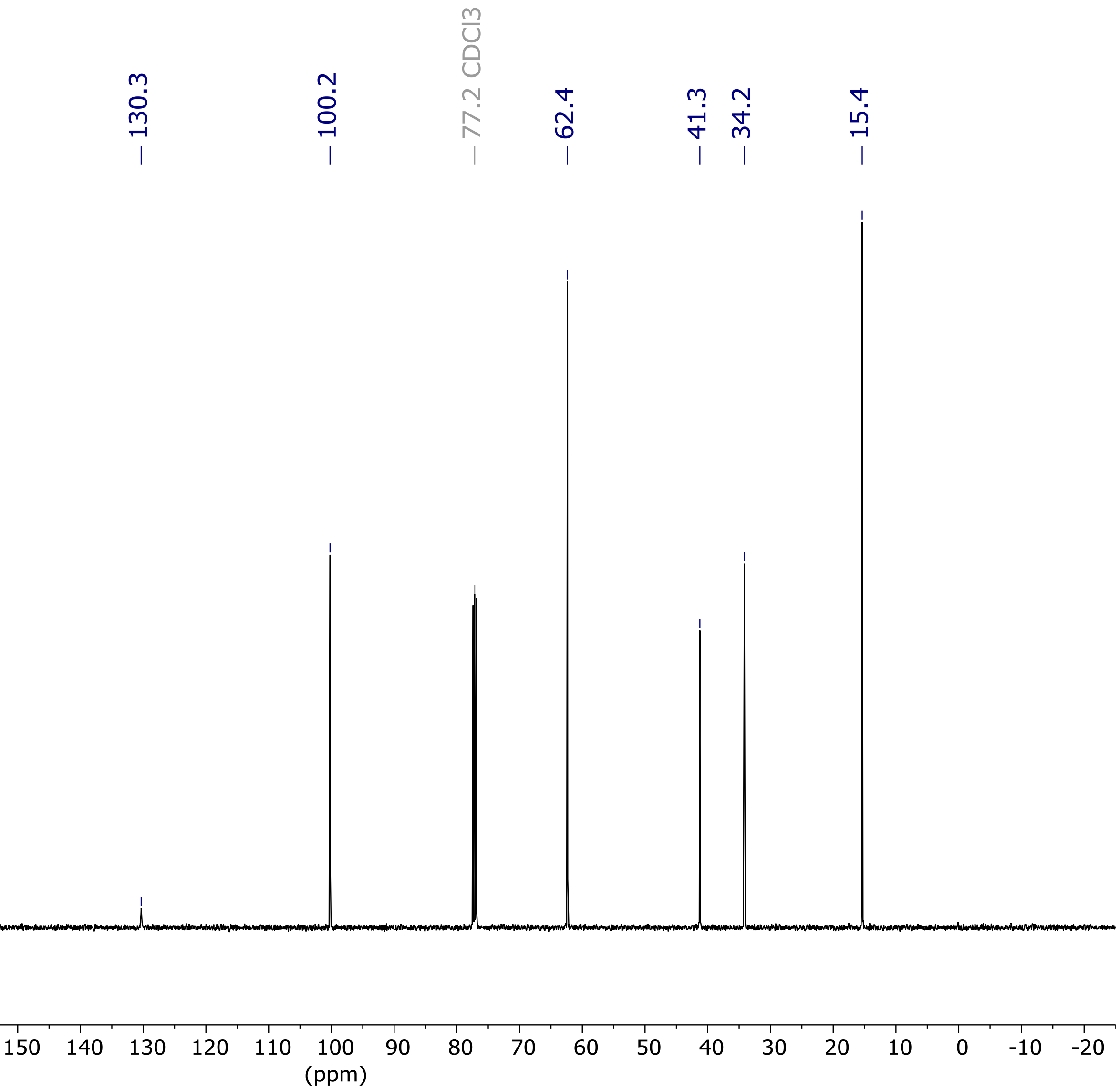
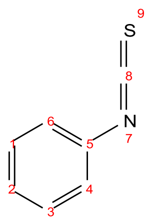
1.20
1.19
1.18
500 MHz, CDCl3
2q
0.93
4.03
2.03
2.00
5.96
(ppm)



126 MHz, $CDCl_3$

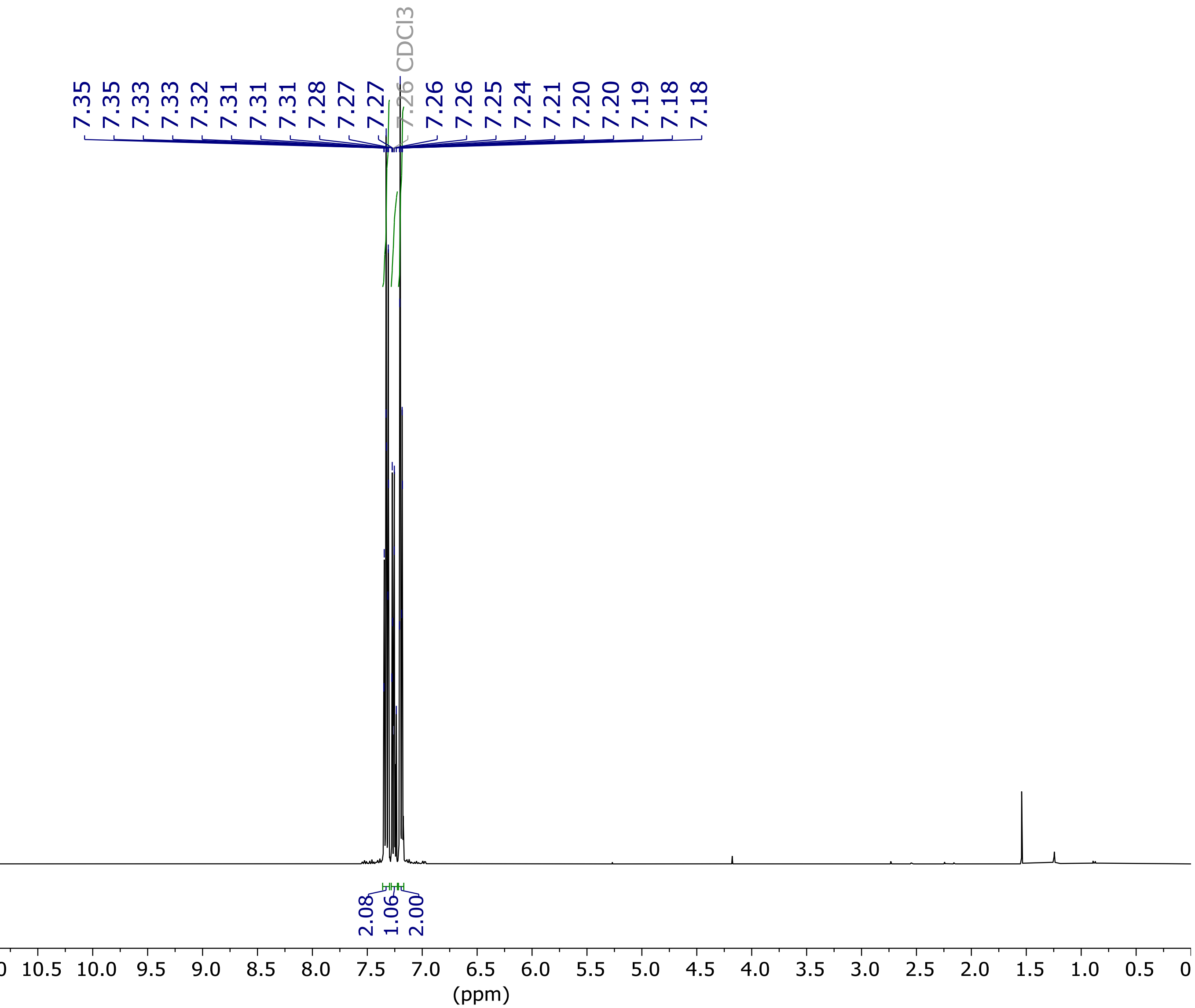
2q

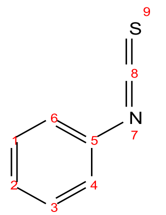




400 MHz, $CDCl_3$

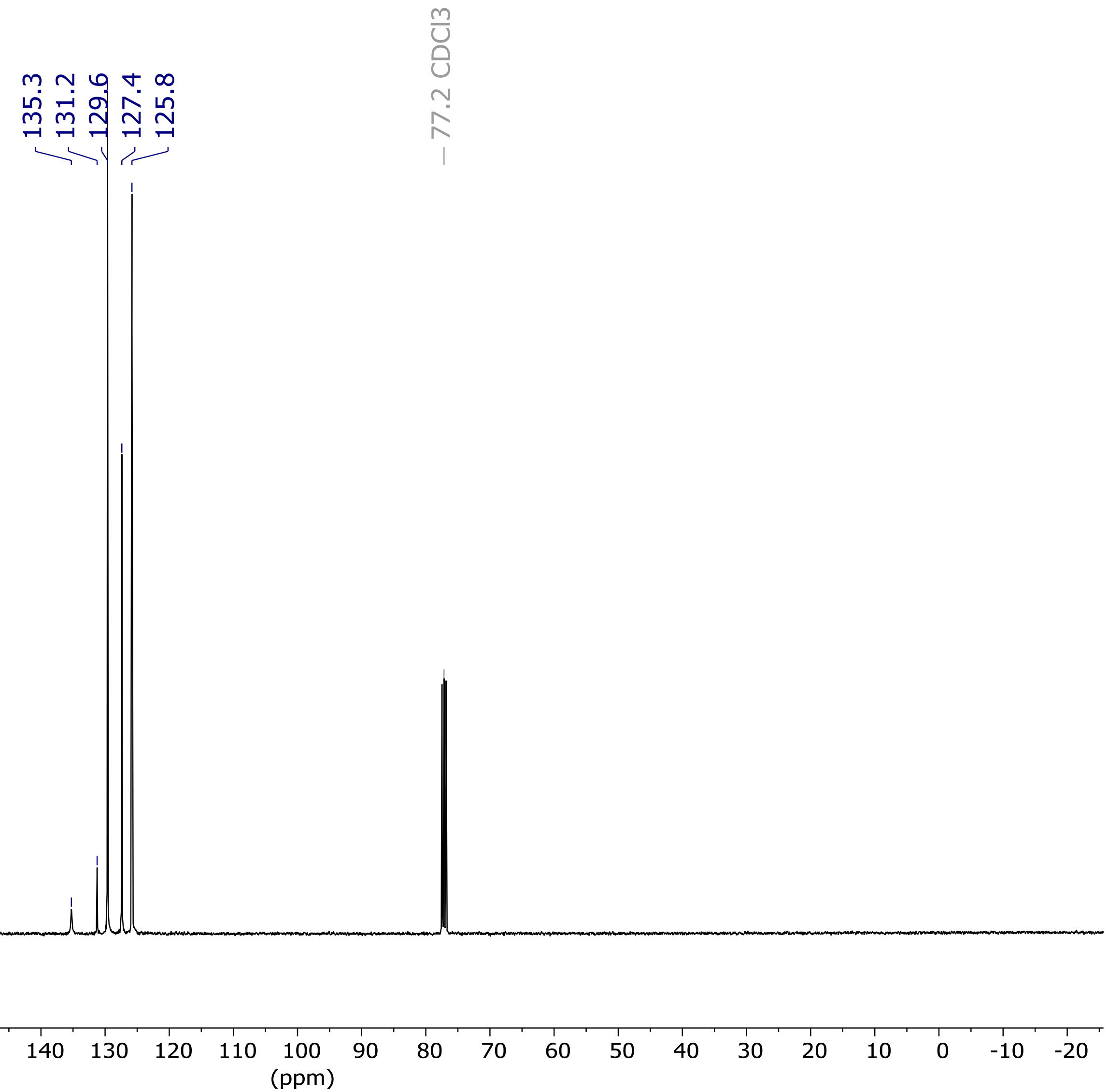
4a

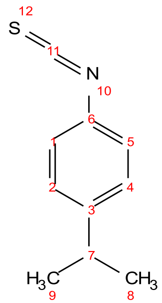




101 MHz, $CDCl_3$

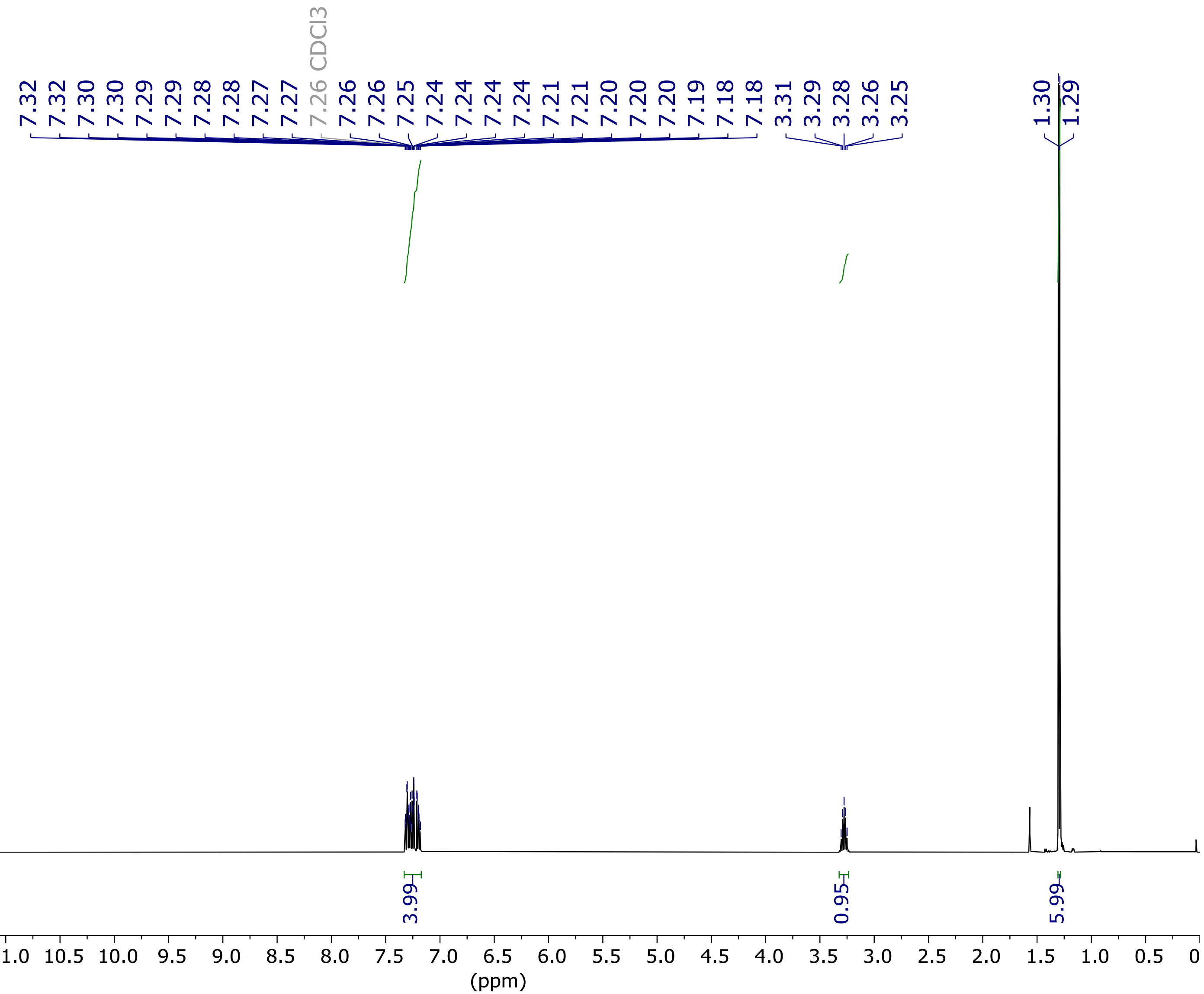
4a

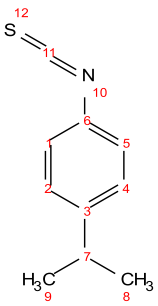




400 MHz, $CDCl_3$

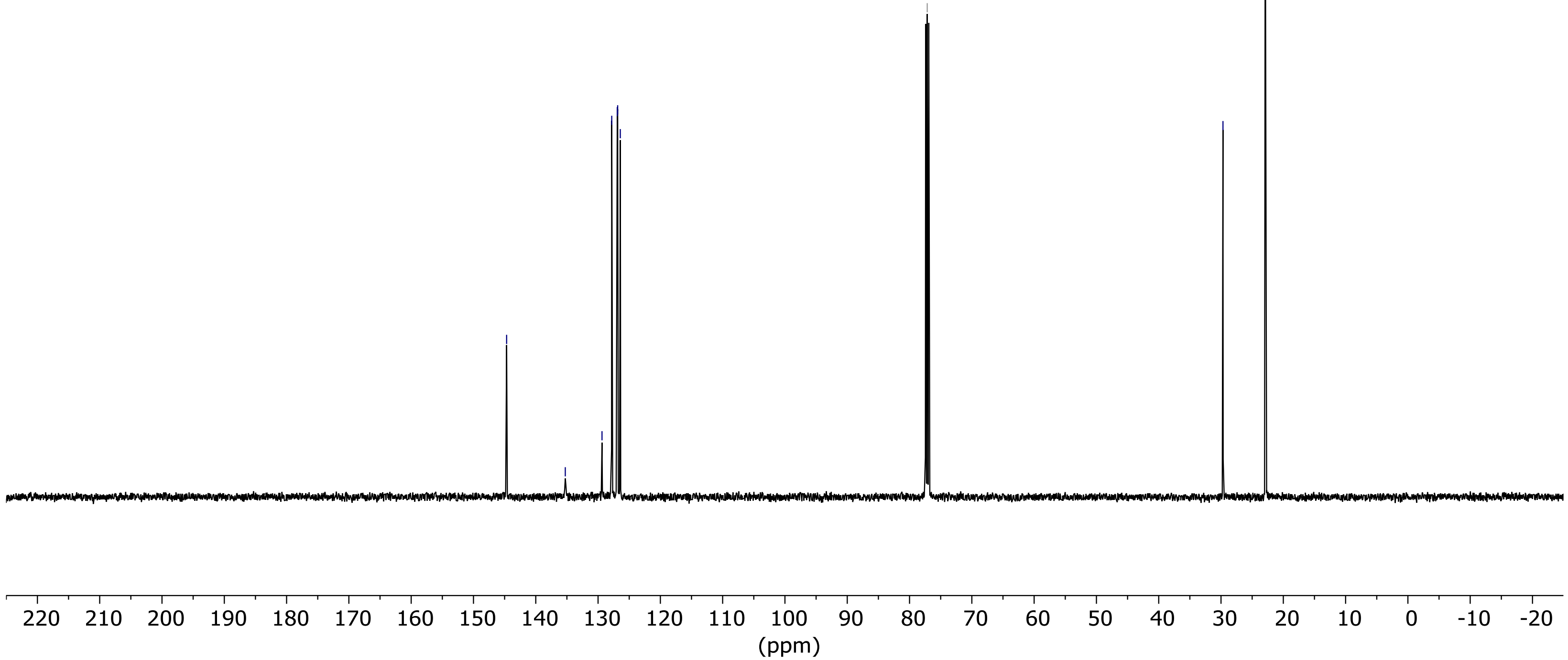
4b

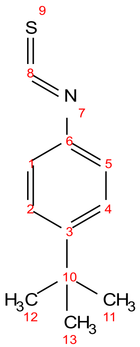




101 MHz, $CDCl_3$

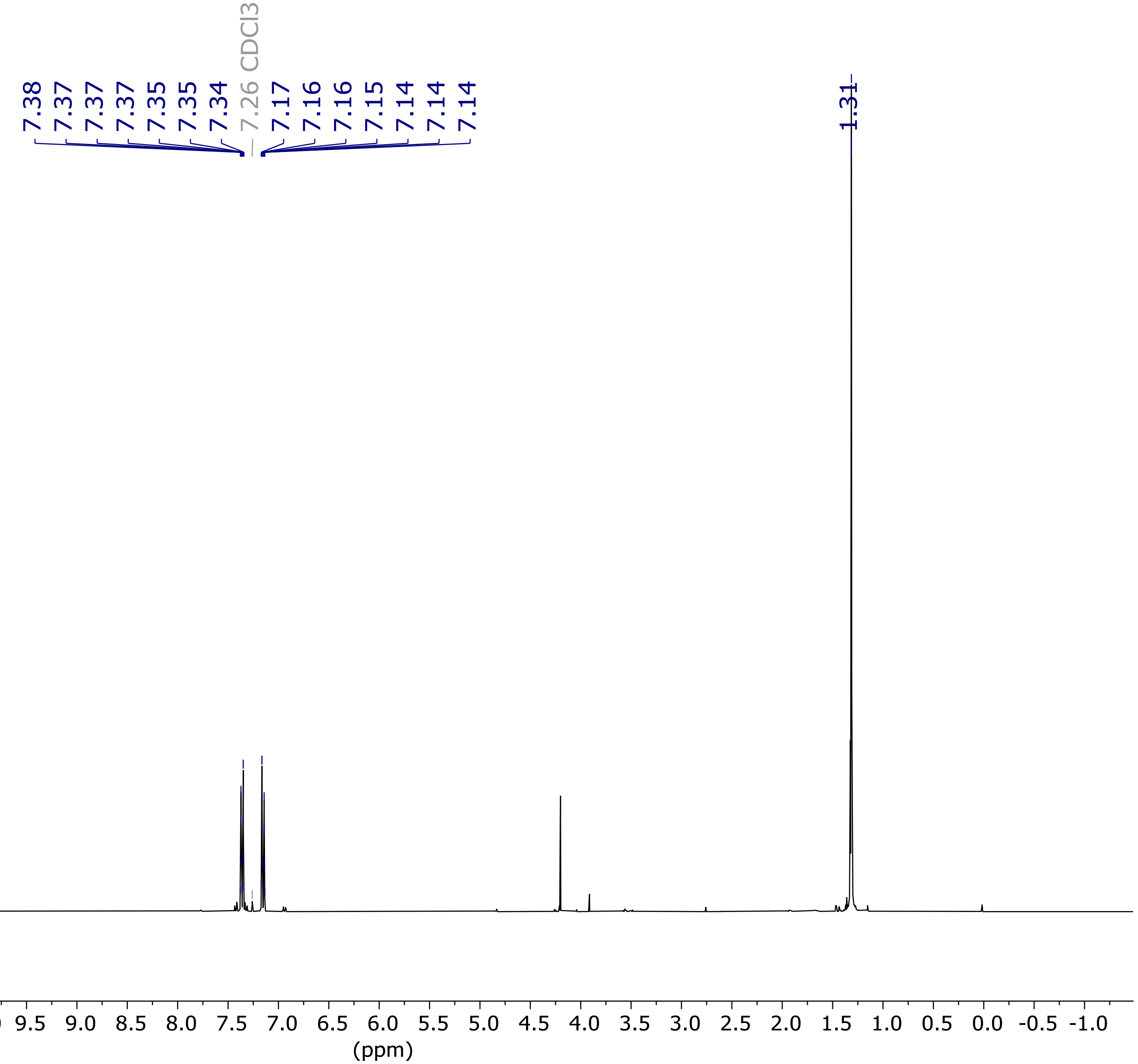
4b

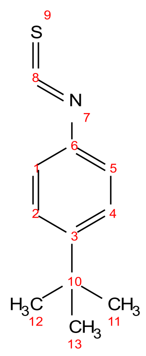




400 MHz, $CDCl_3$

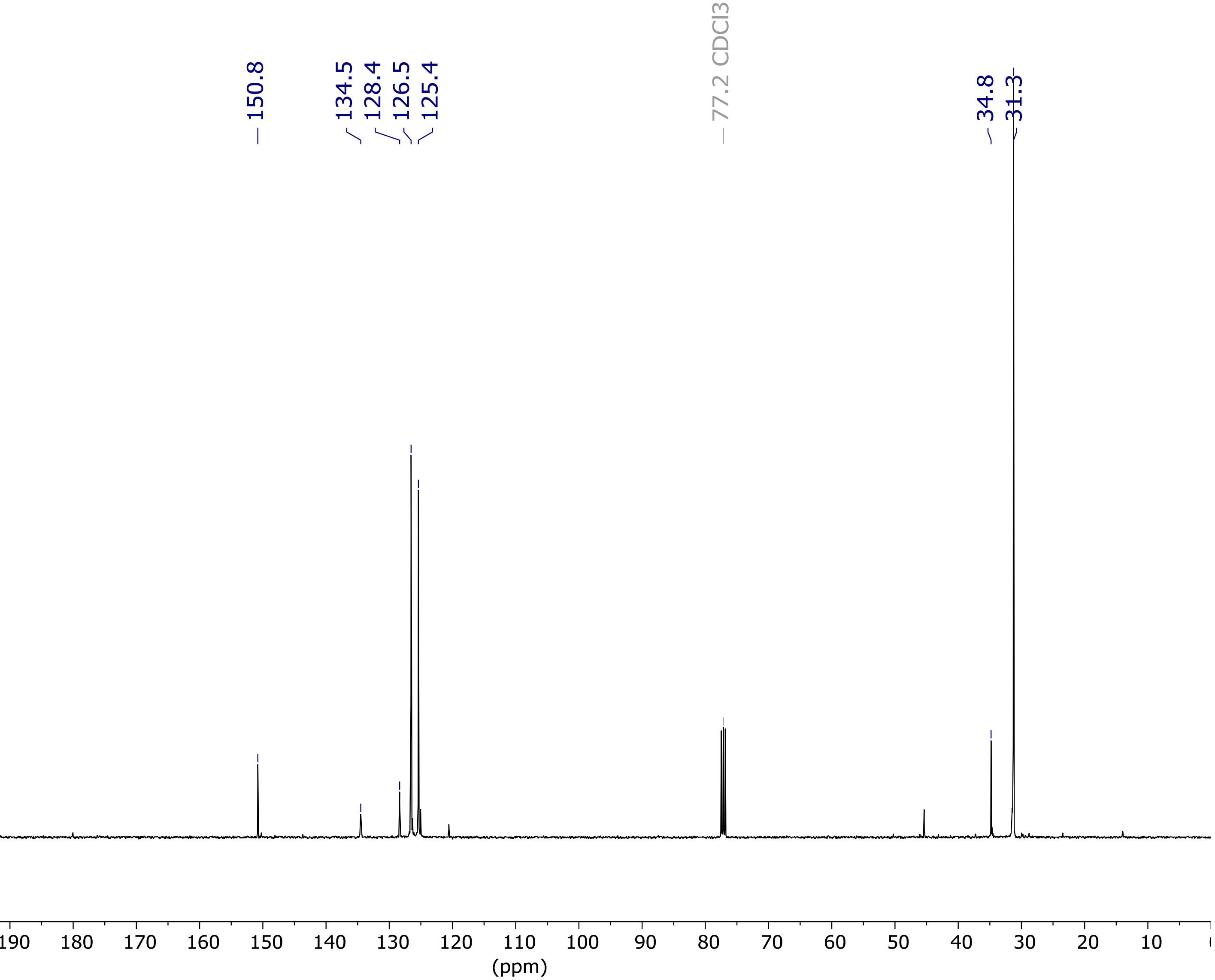
4c

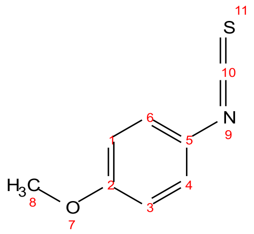




101 MHz, $CDCl_3$

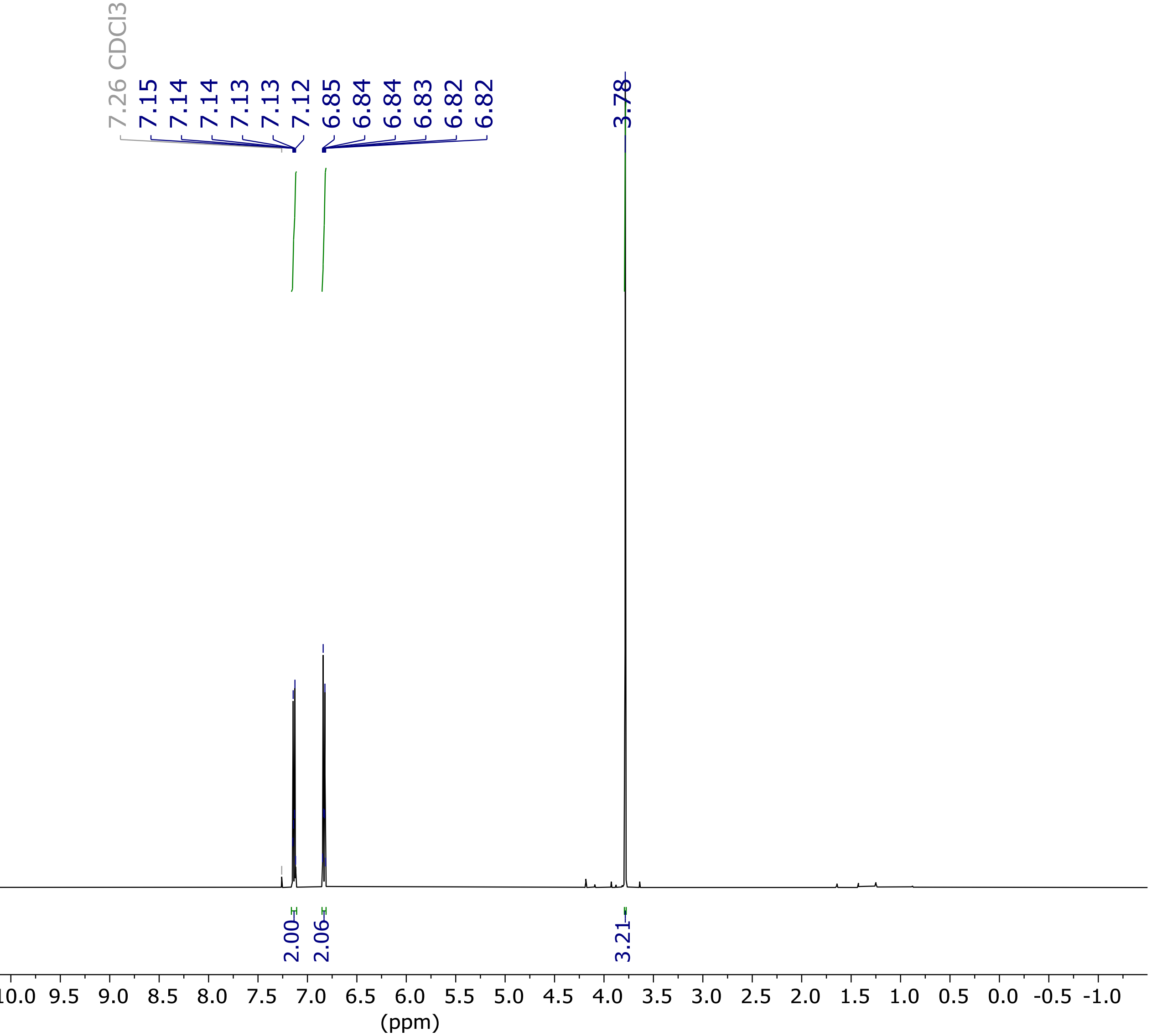
4c

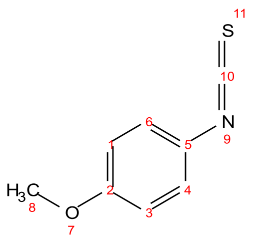




400 MHz, $CDCl_3$

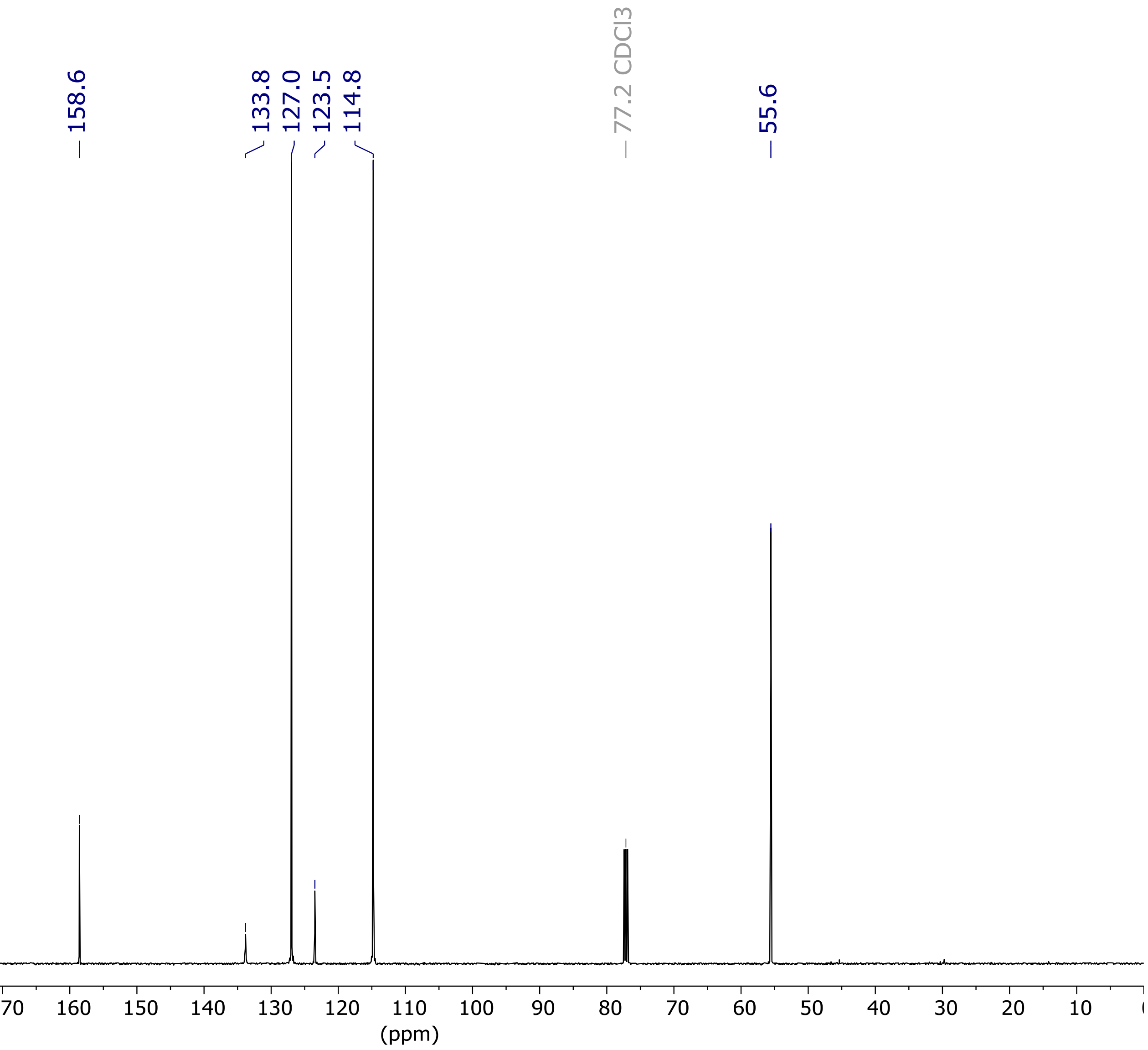
4d

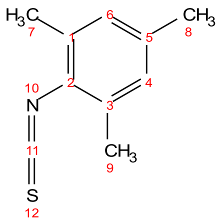




101 MHz, $CDCl_3$

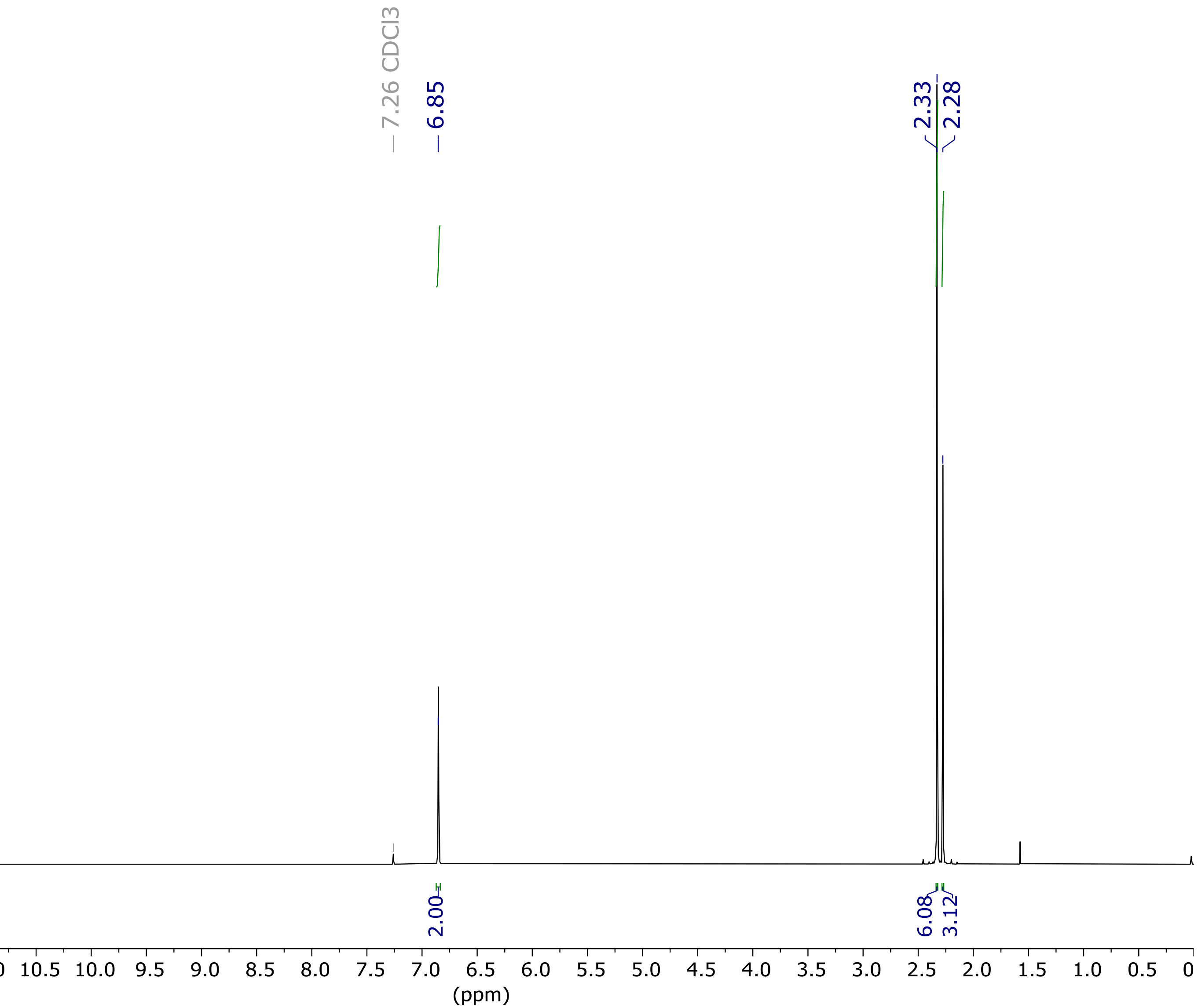
4d

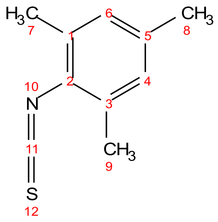




400 MHz, $CDCl_3$

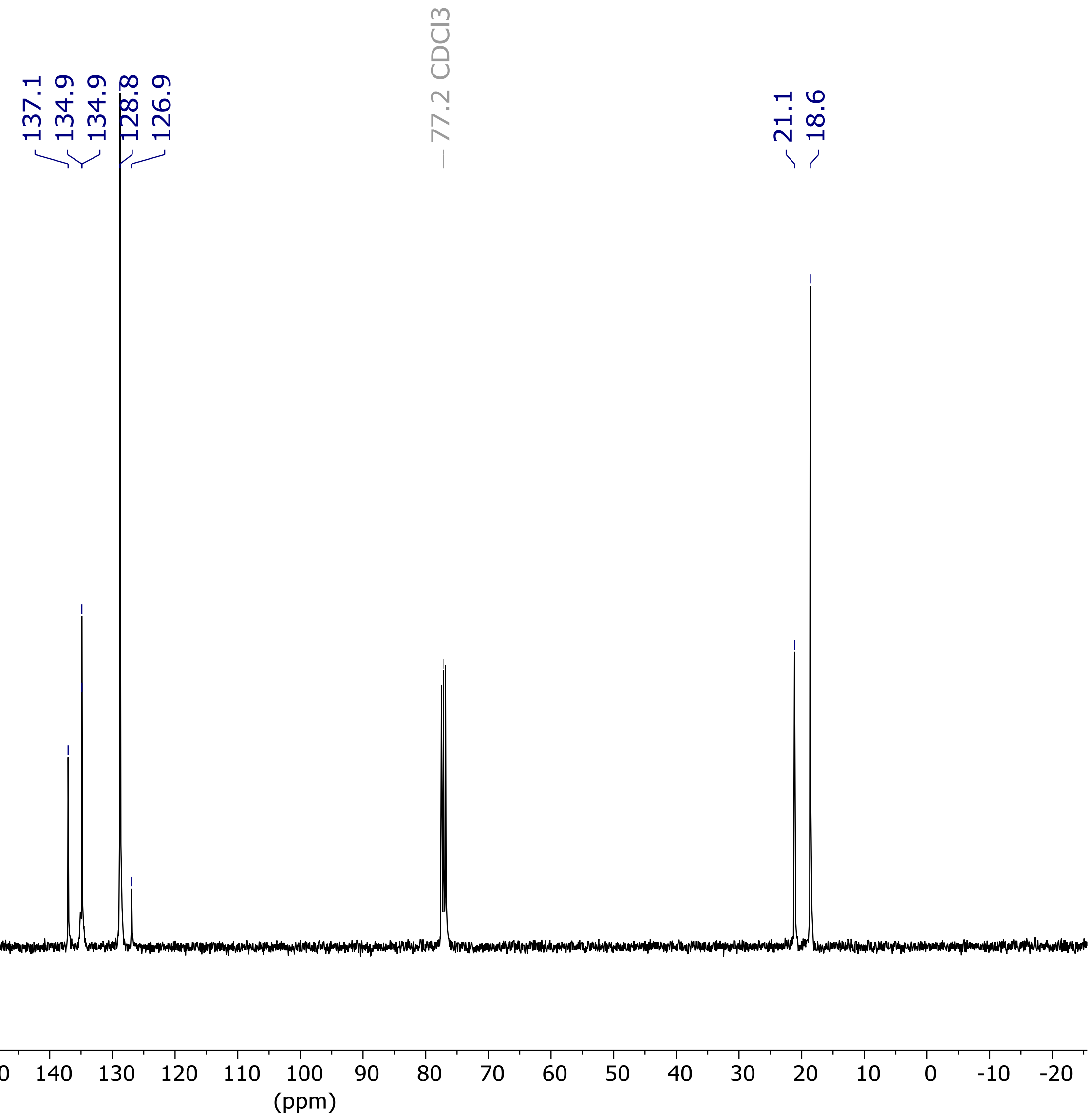
4e

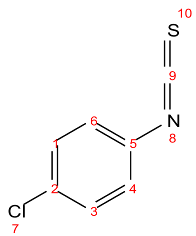




101 MHz, $CDCl_3$

4e





400 MHz, $CDCl_3$

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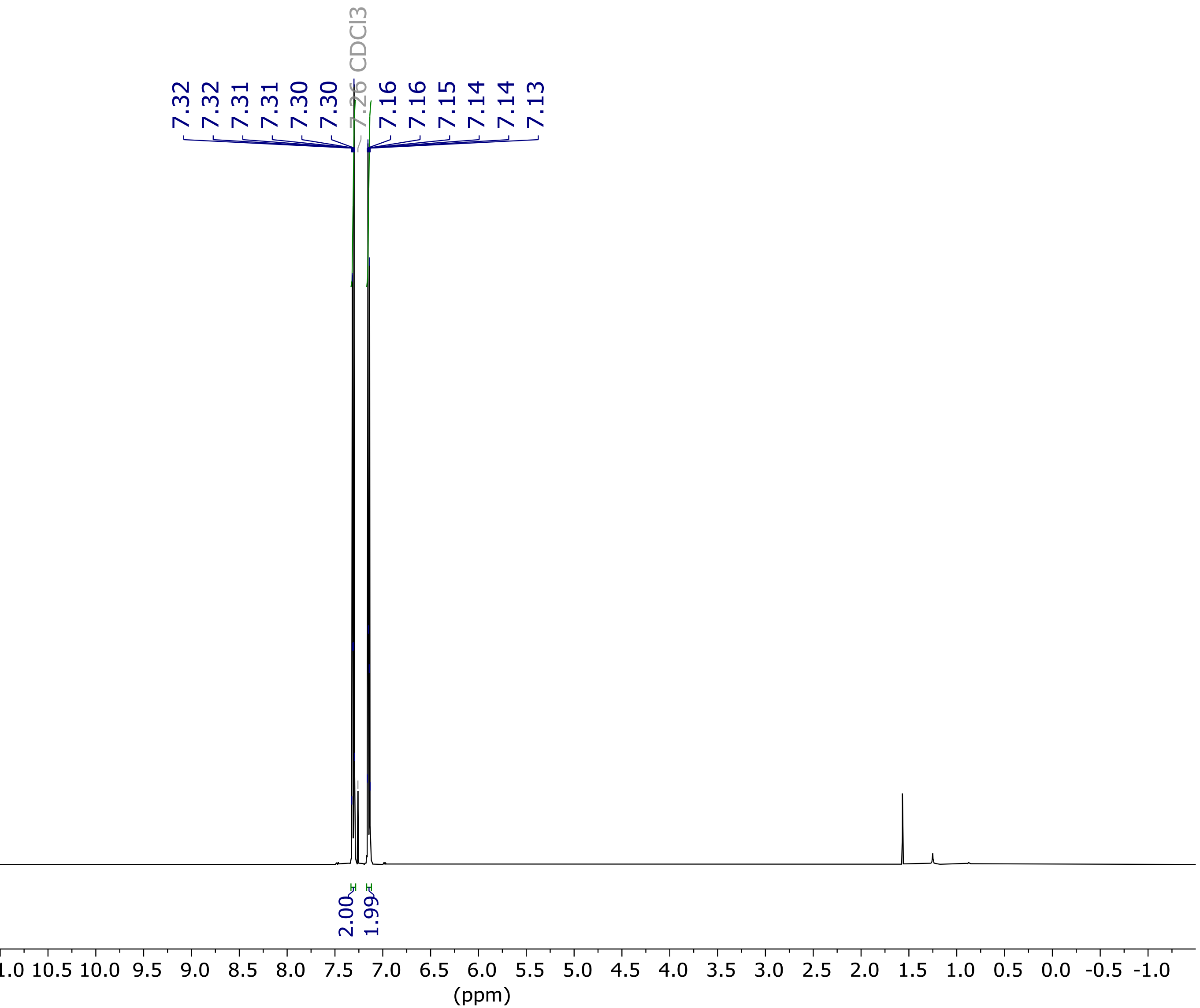

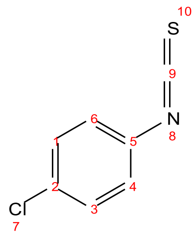

101 MHz, $CDCl_3$

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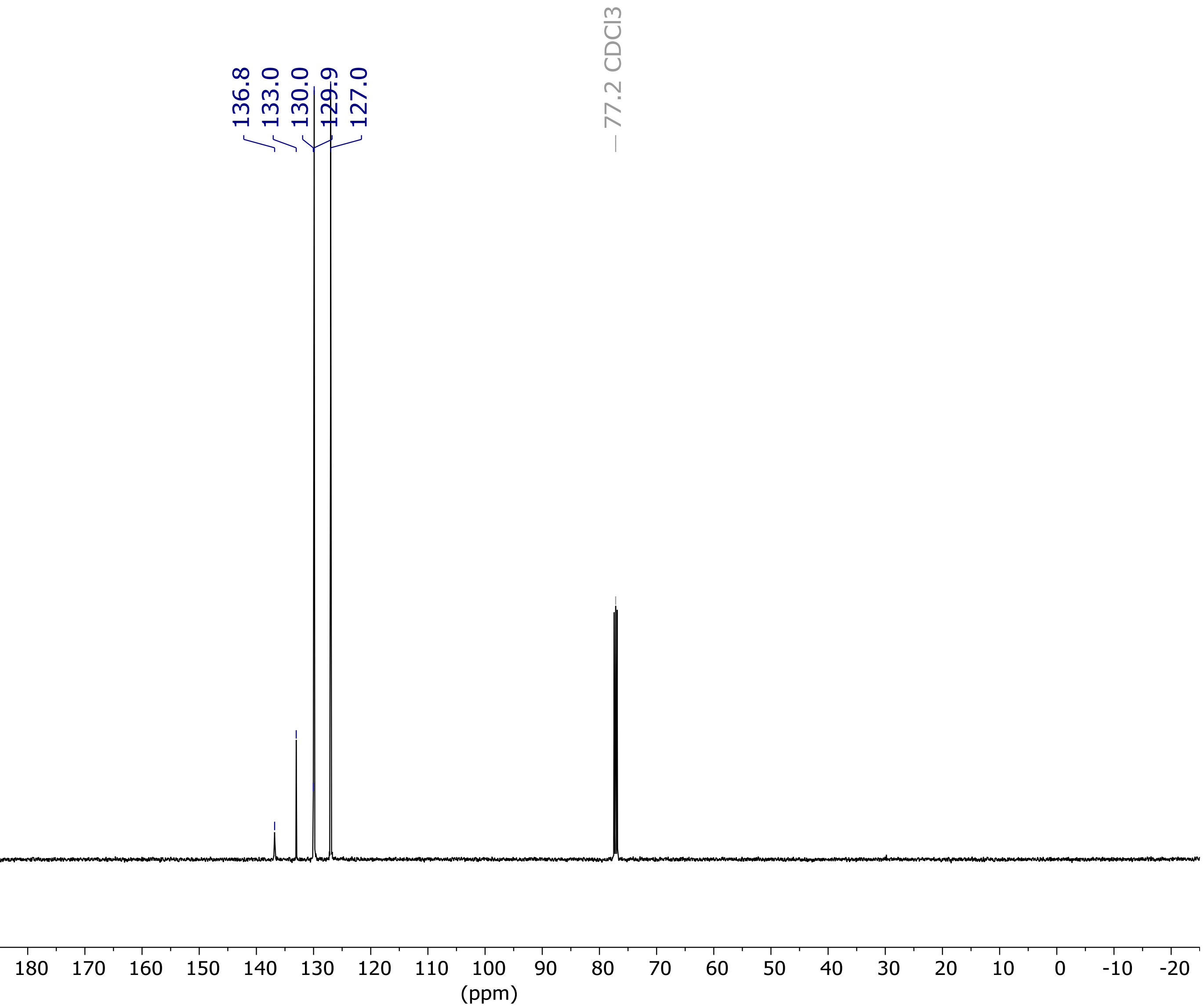
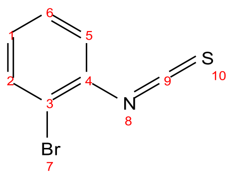
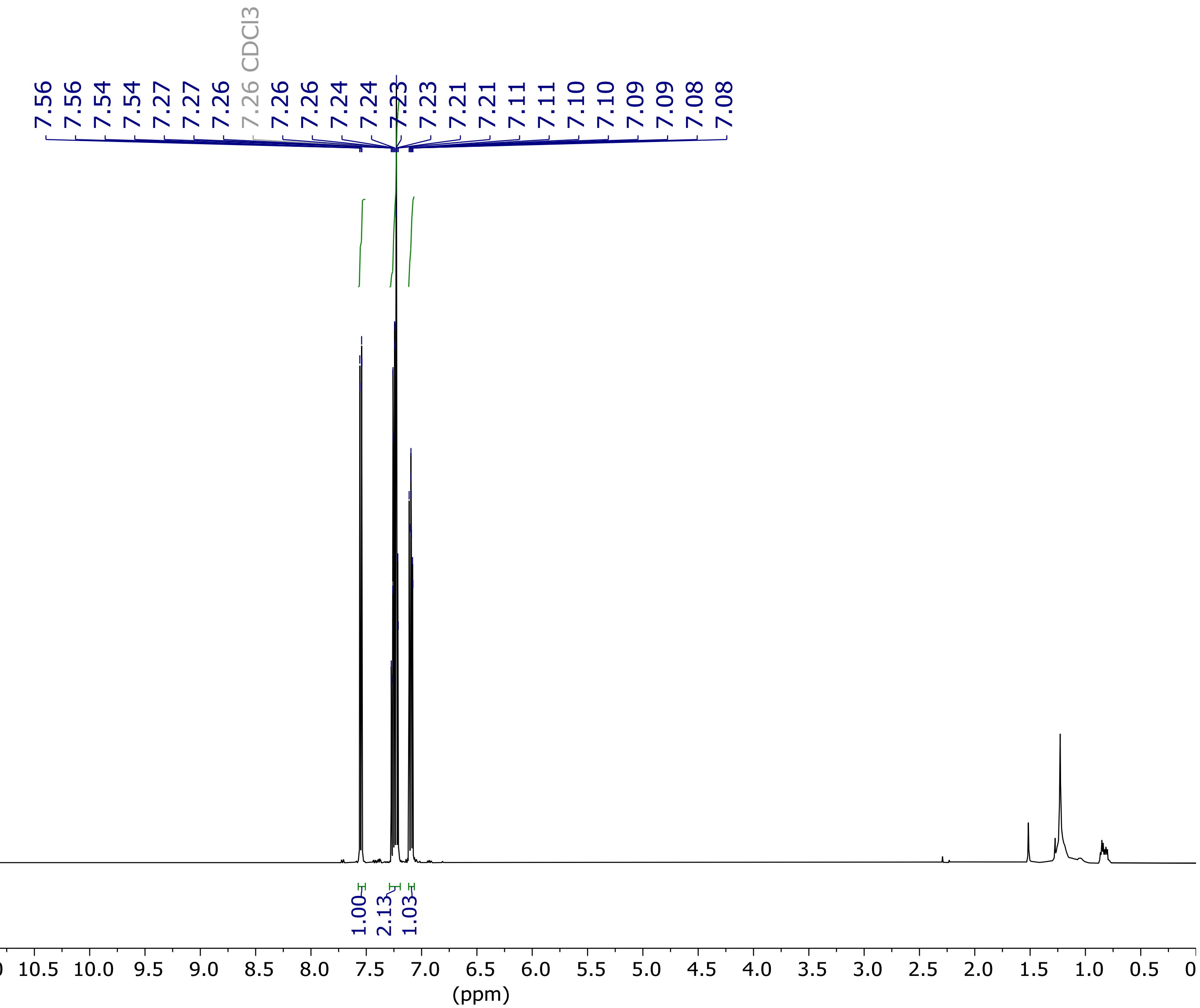
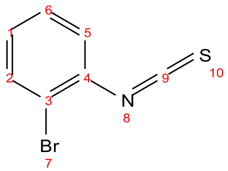




400 MHz, $CDCl_3$

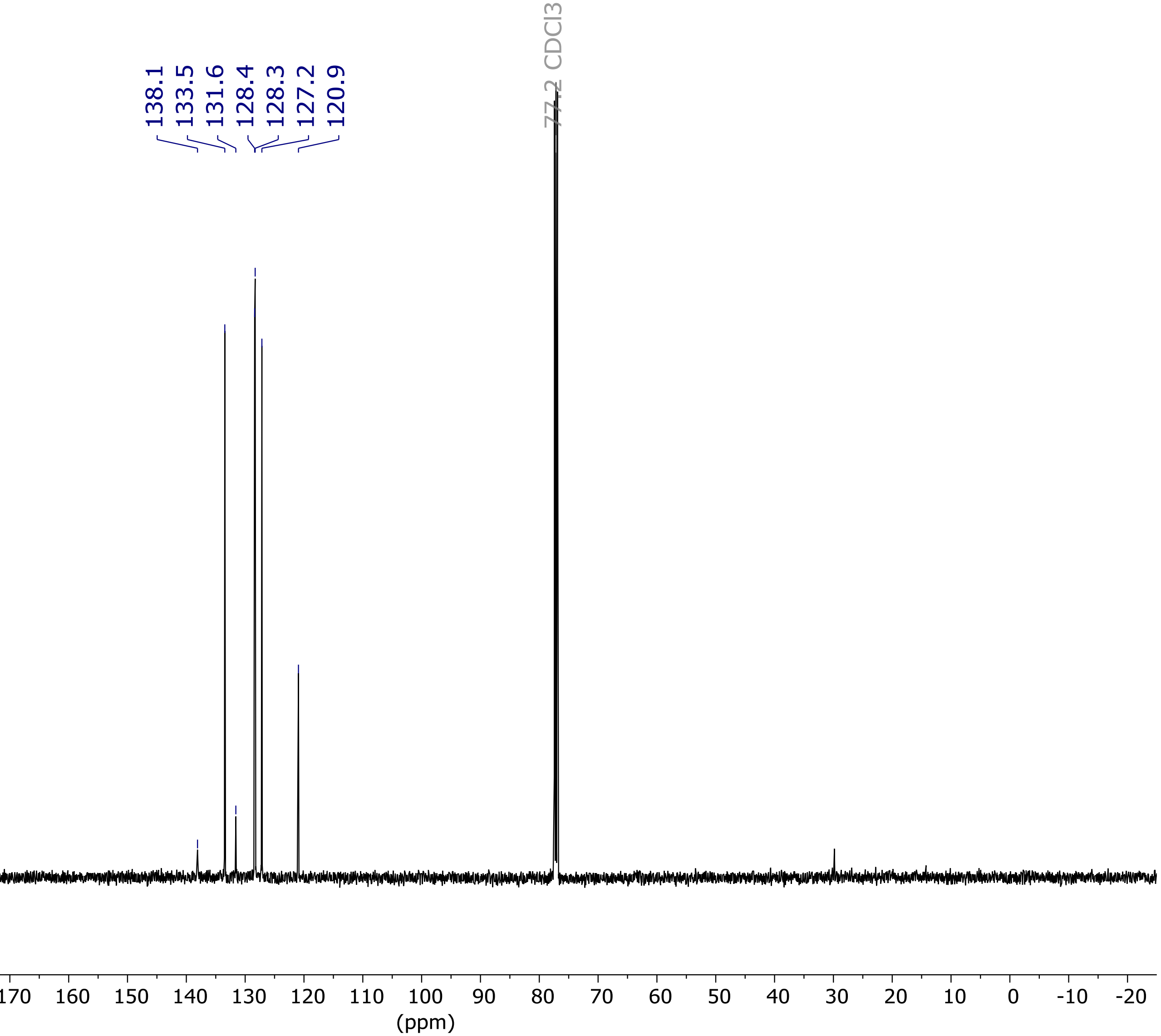
4g

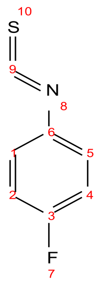




101 MHz, $CDCl_3$

4g





400 MHz, $CDCl_3$

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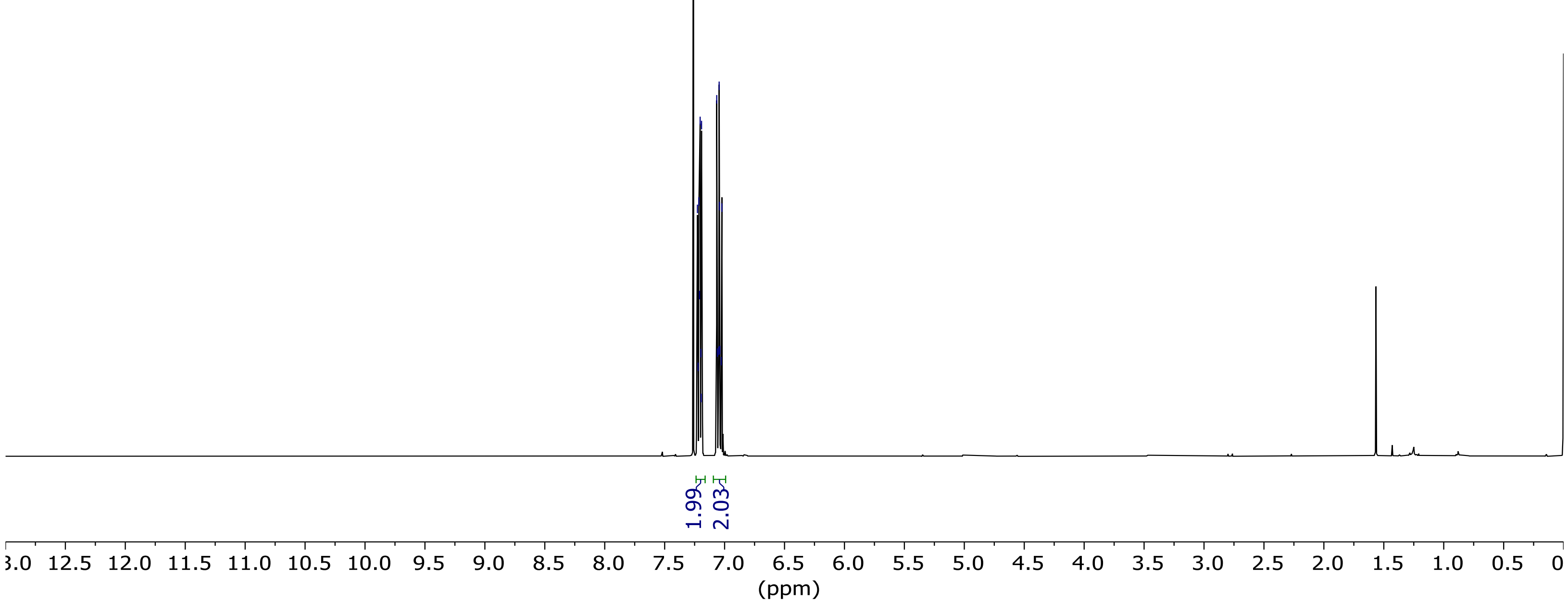
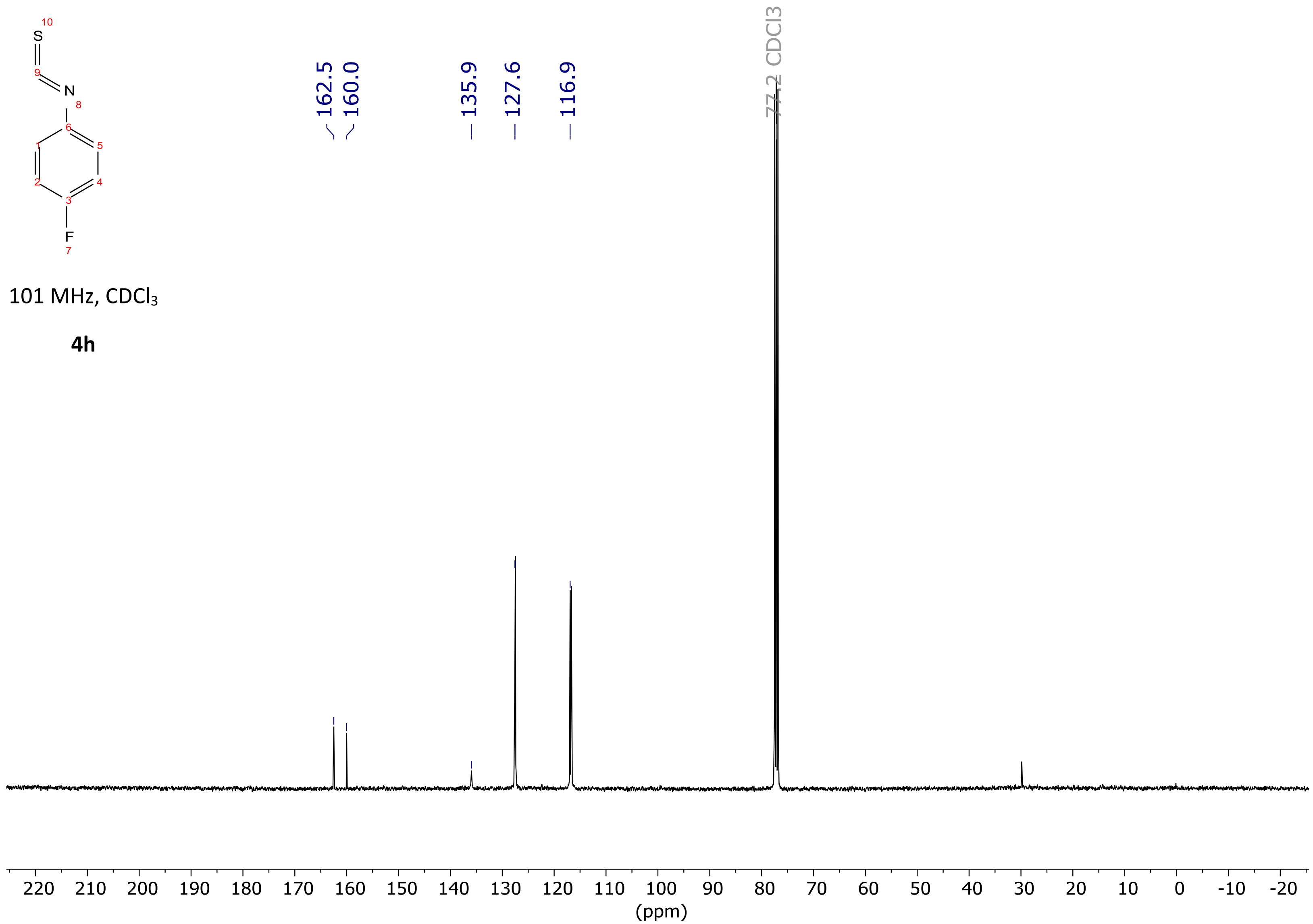
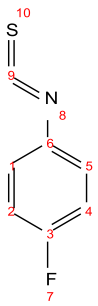




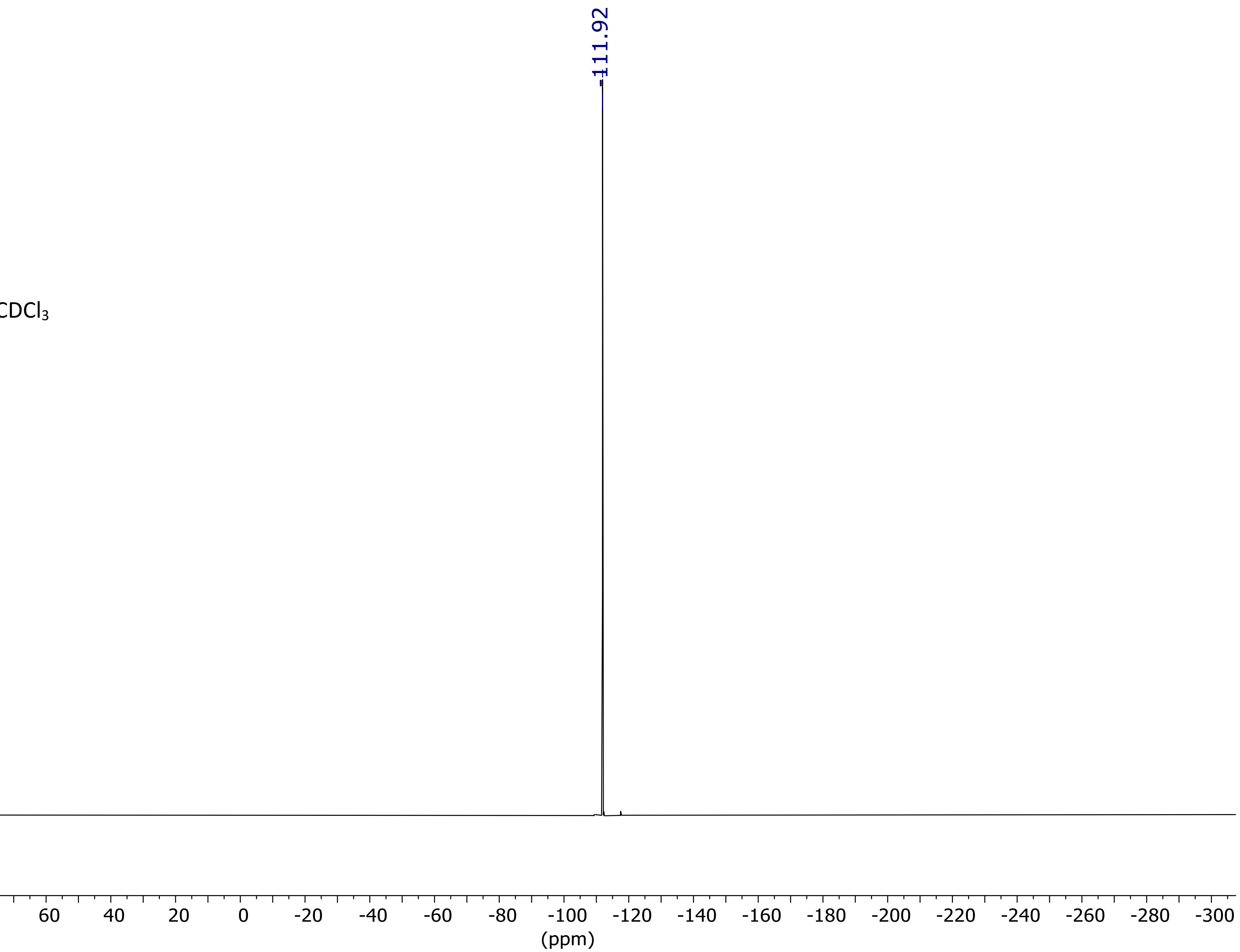
101 MHz, $CDCl_3$

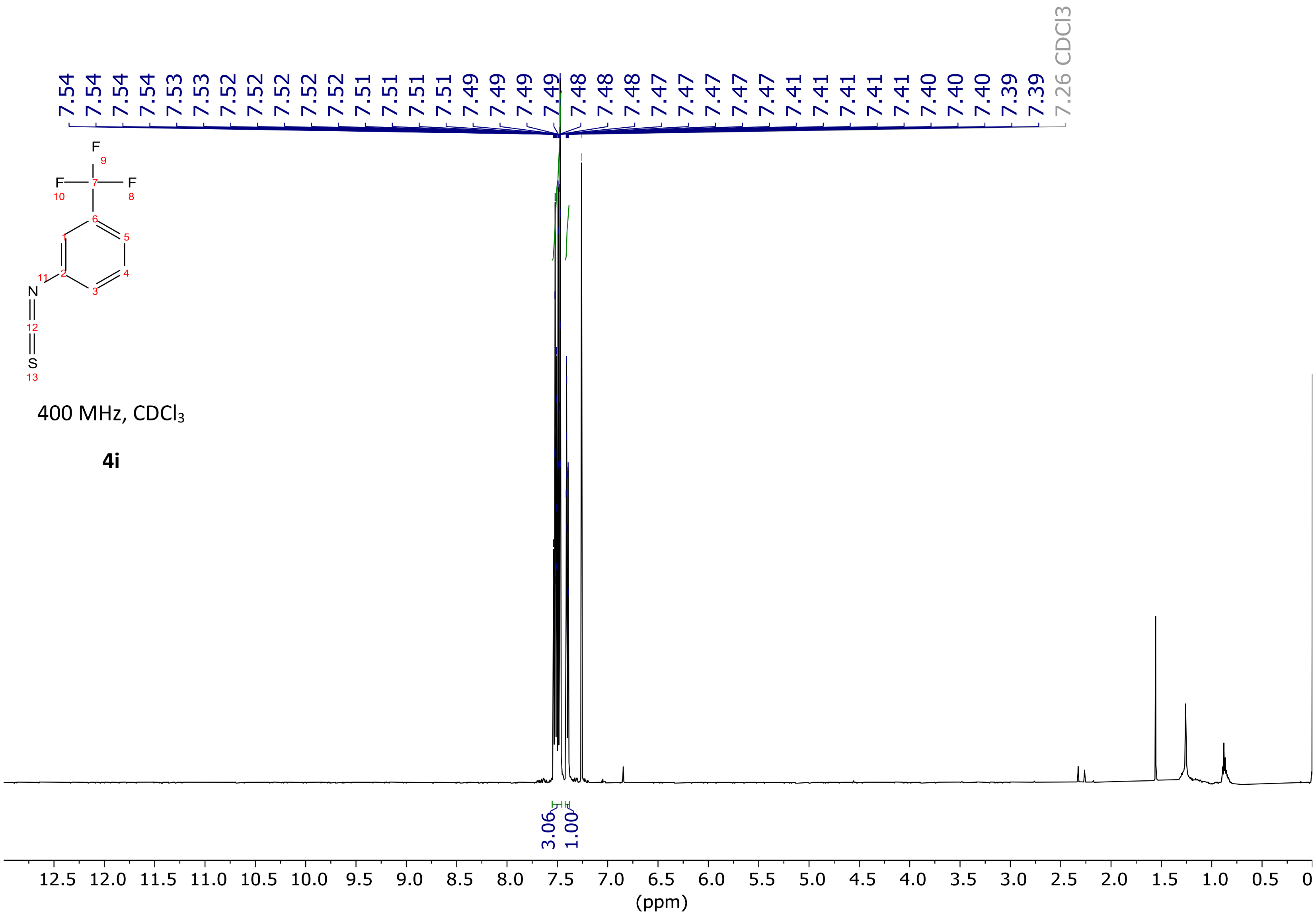
4h



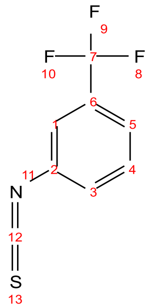
376 MHz, $CDCl_3$

4h



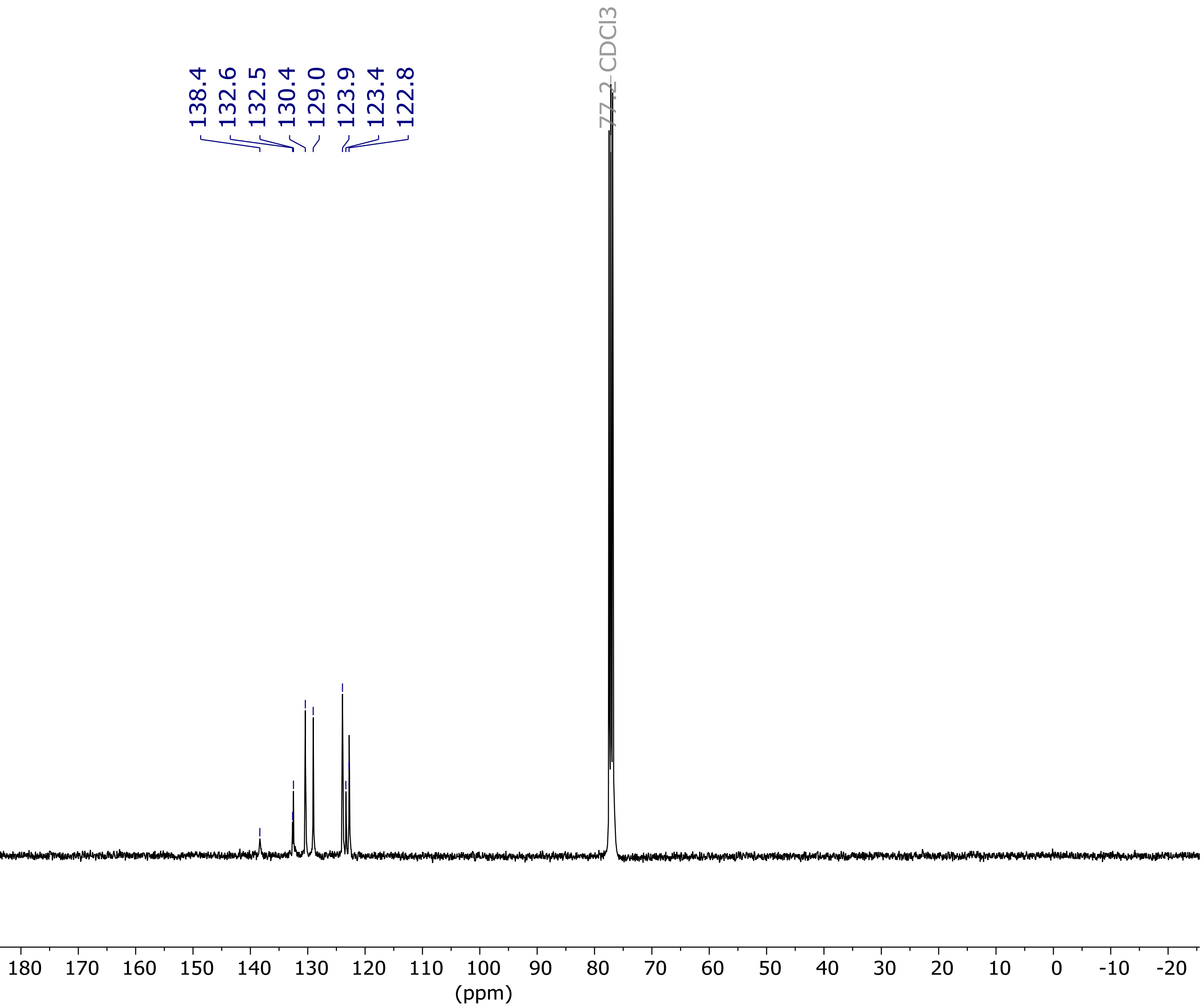

7.54 7.54 7.54 7.54 7.53 7.53 7.52 7.52 7.52 7.52 7.52 7.51 7.51 7.51 7.51 7.49 7.49 7.49 7.49 7.48 7.48 7.48 7.47 7.47 7.47 7.47 7.47 7.41 7.41 7.41 7.41 7.41 7.40 7.40 7.40 7.39 7.39 7.26 CDCl3
400 MHz, $CDCl_3$
3.06
1.00
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0
(ppm)

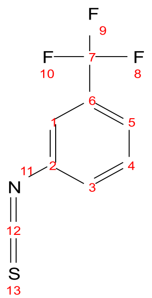
4i



101 MHz, $CDCl_3$

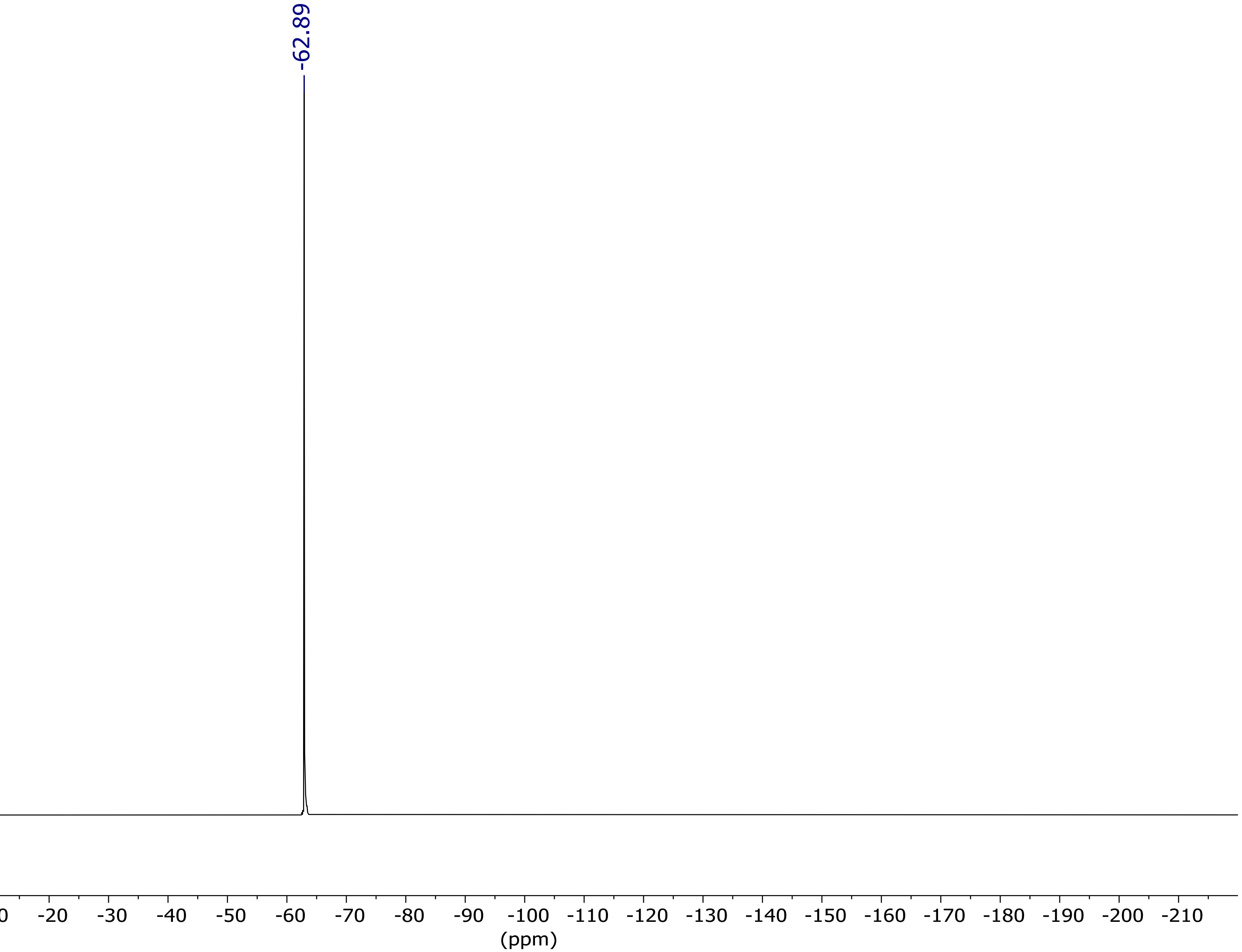
4i

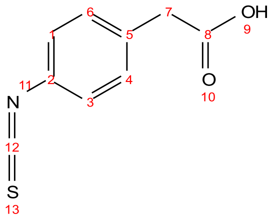




376 MHz, $CDCl_3$

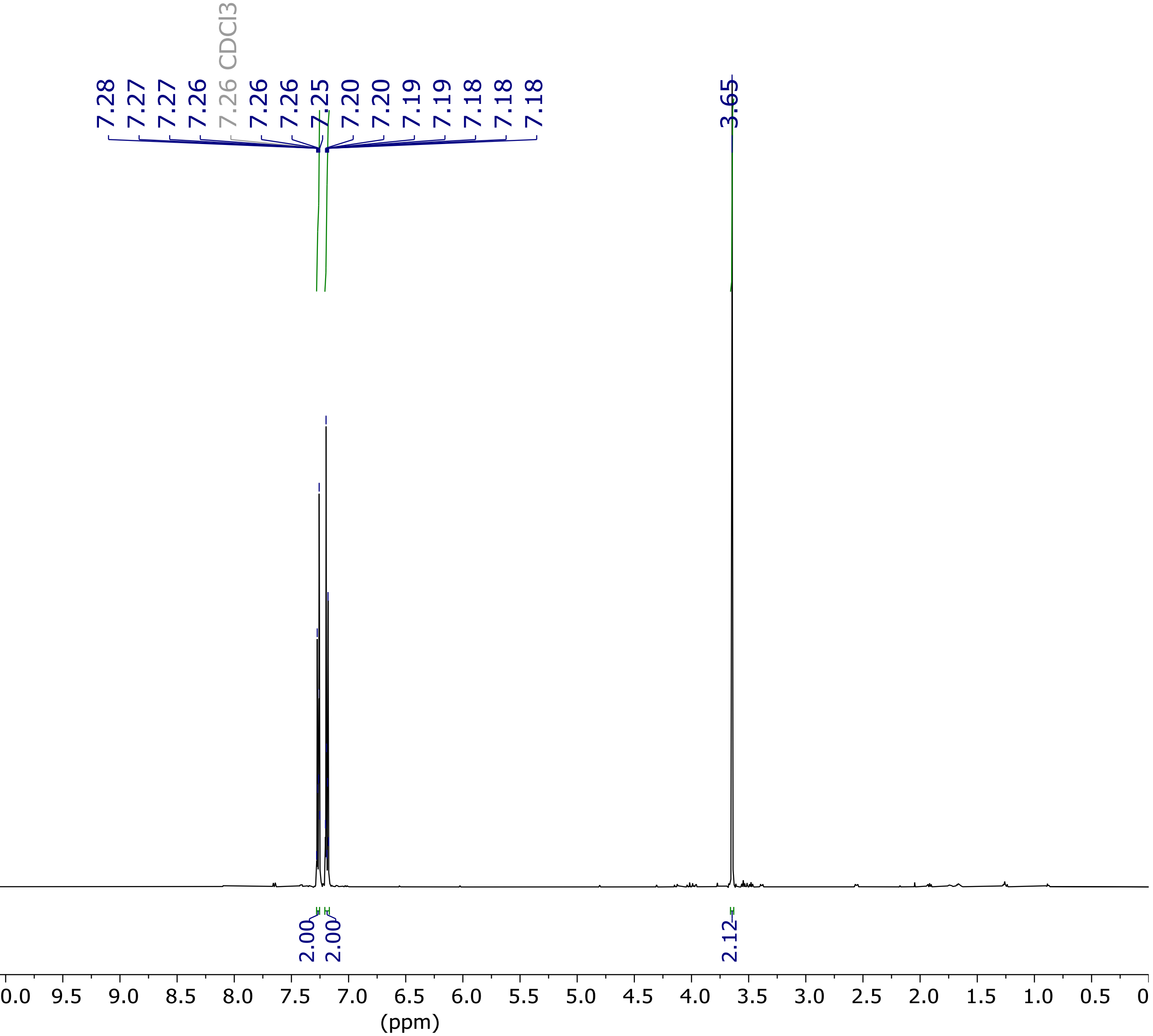
4i

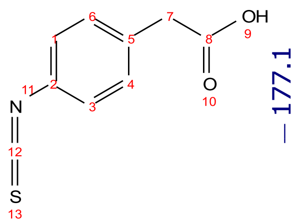




400 MHz, $CDCl_3$

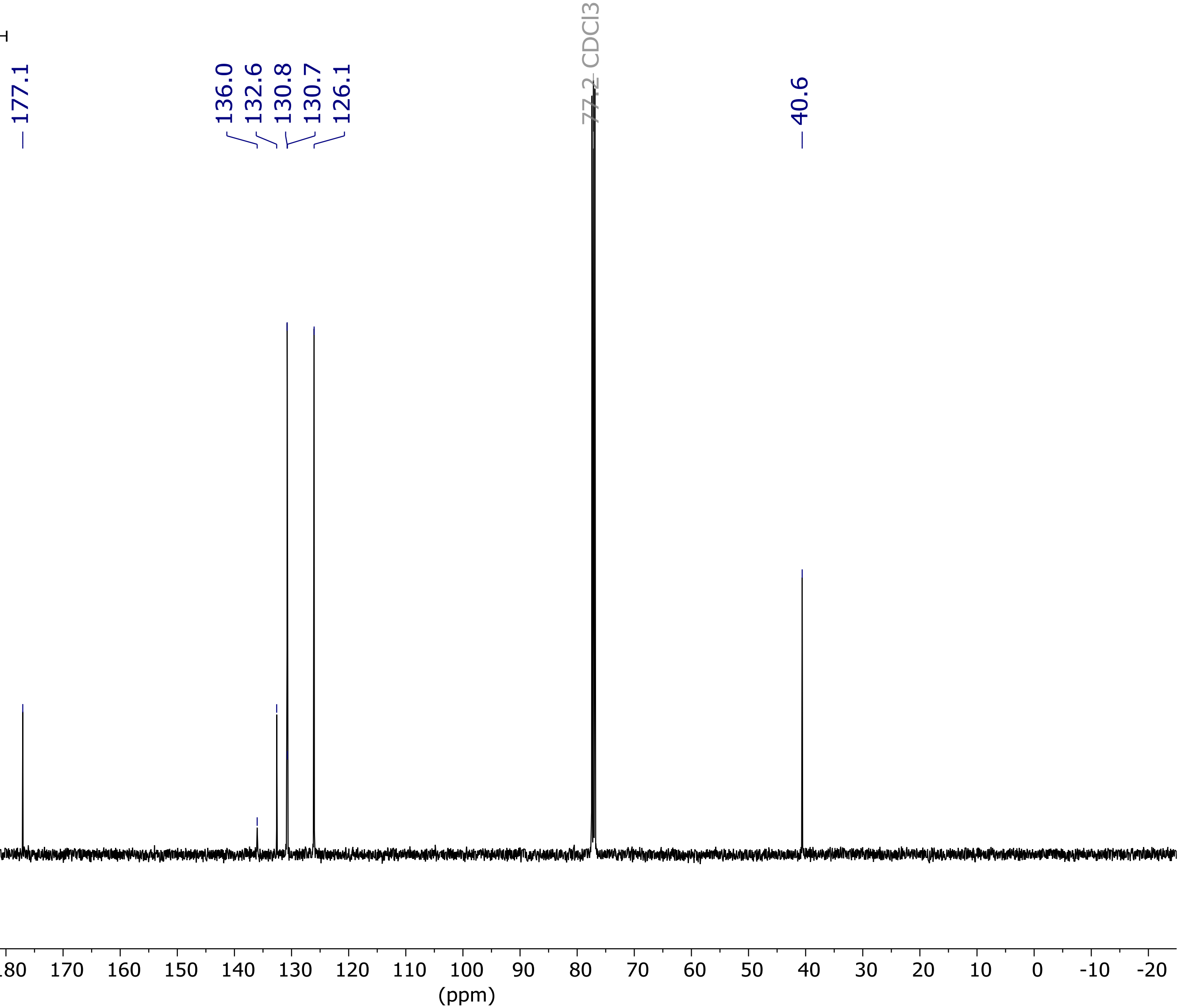
4j

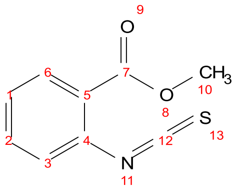




101 MHz, $CDCl_3$

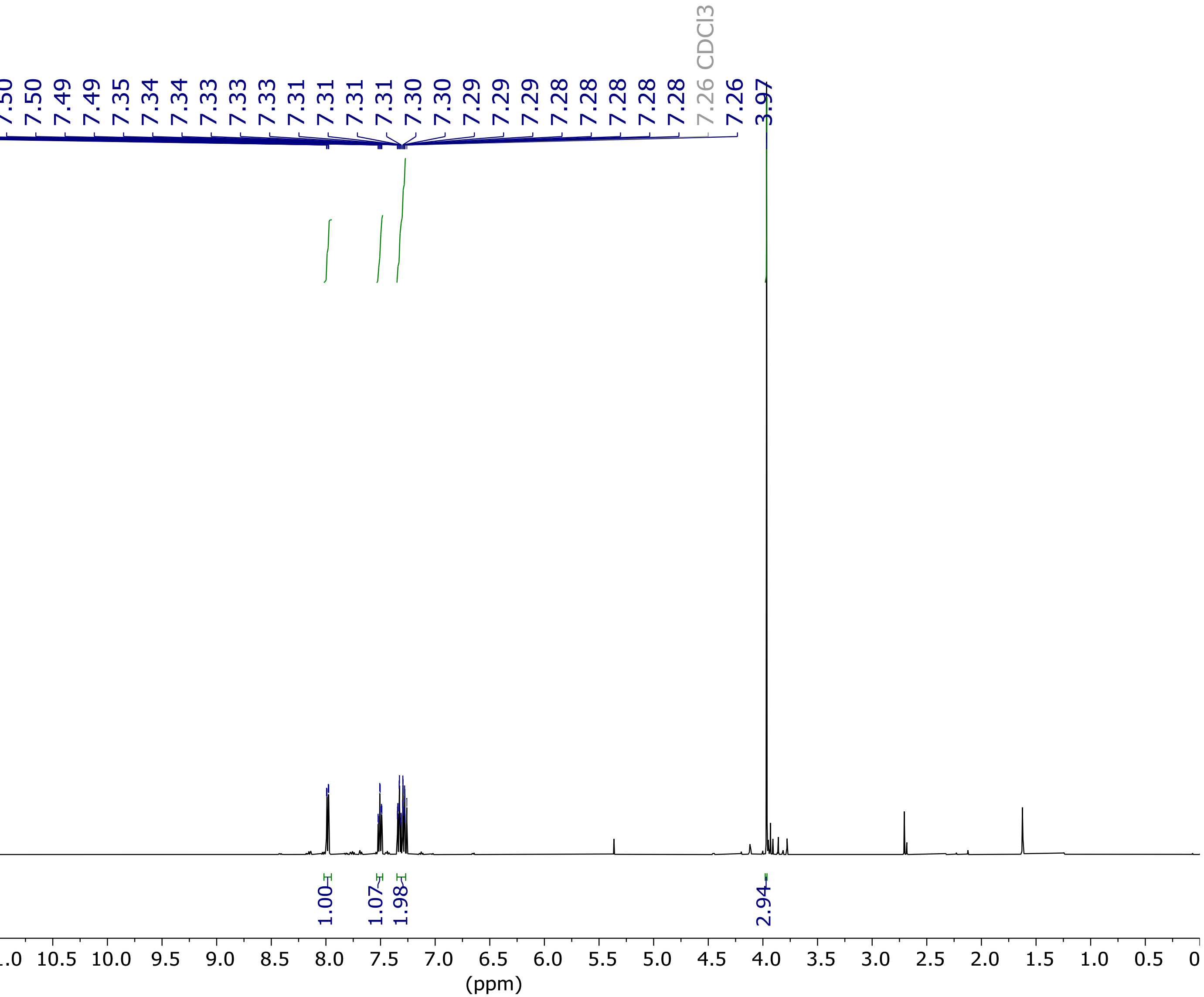
4j

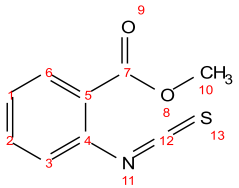




400 MHz, $CDCl_3$

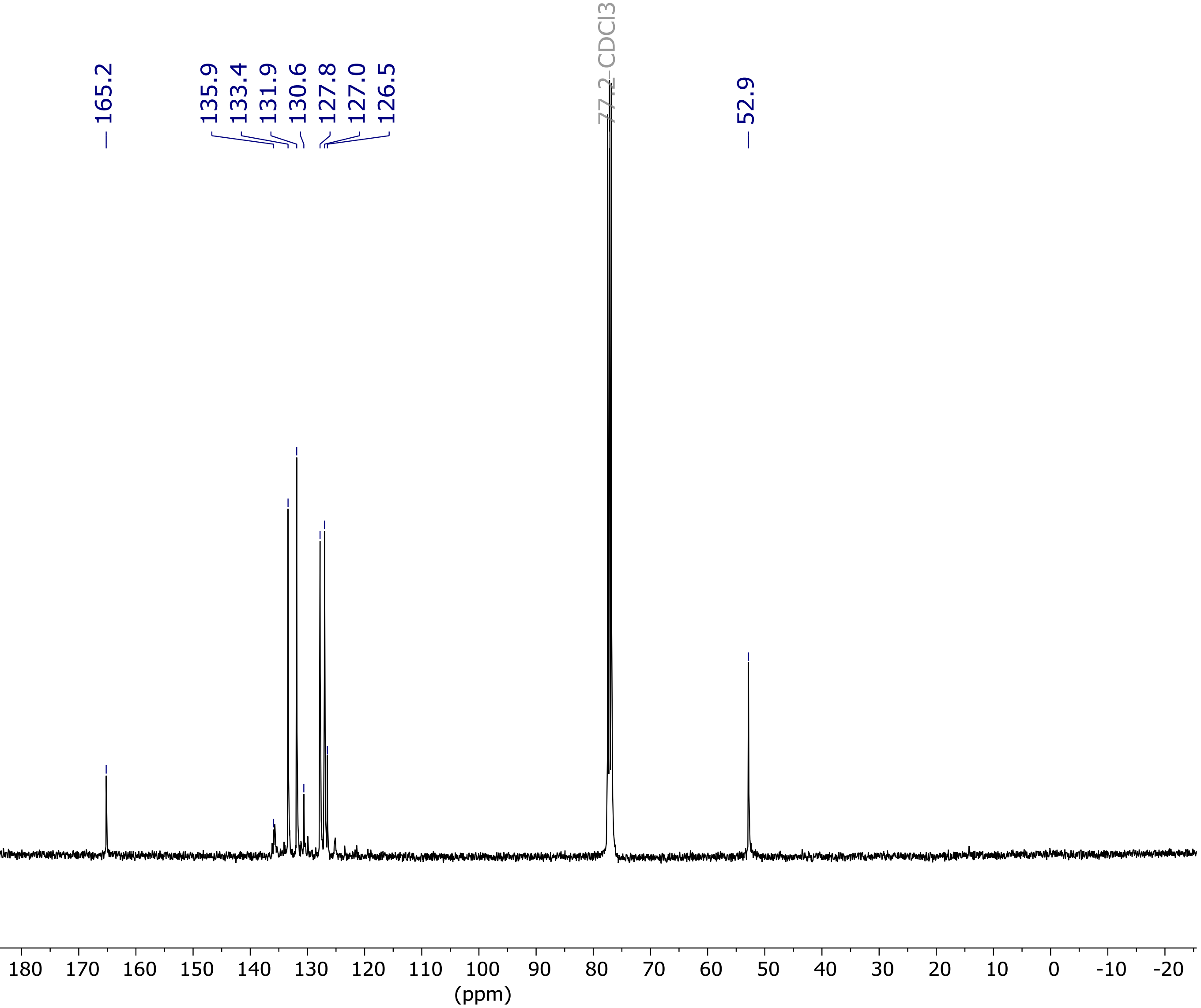
4k

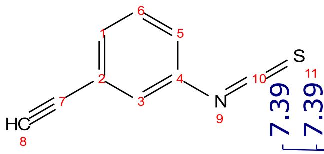




101 MHz, $CDCl_3$

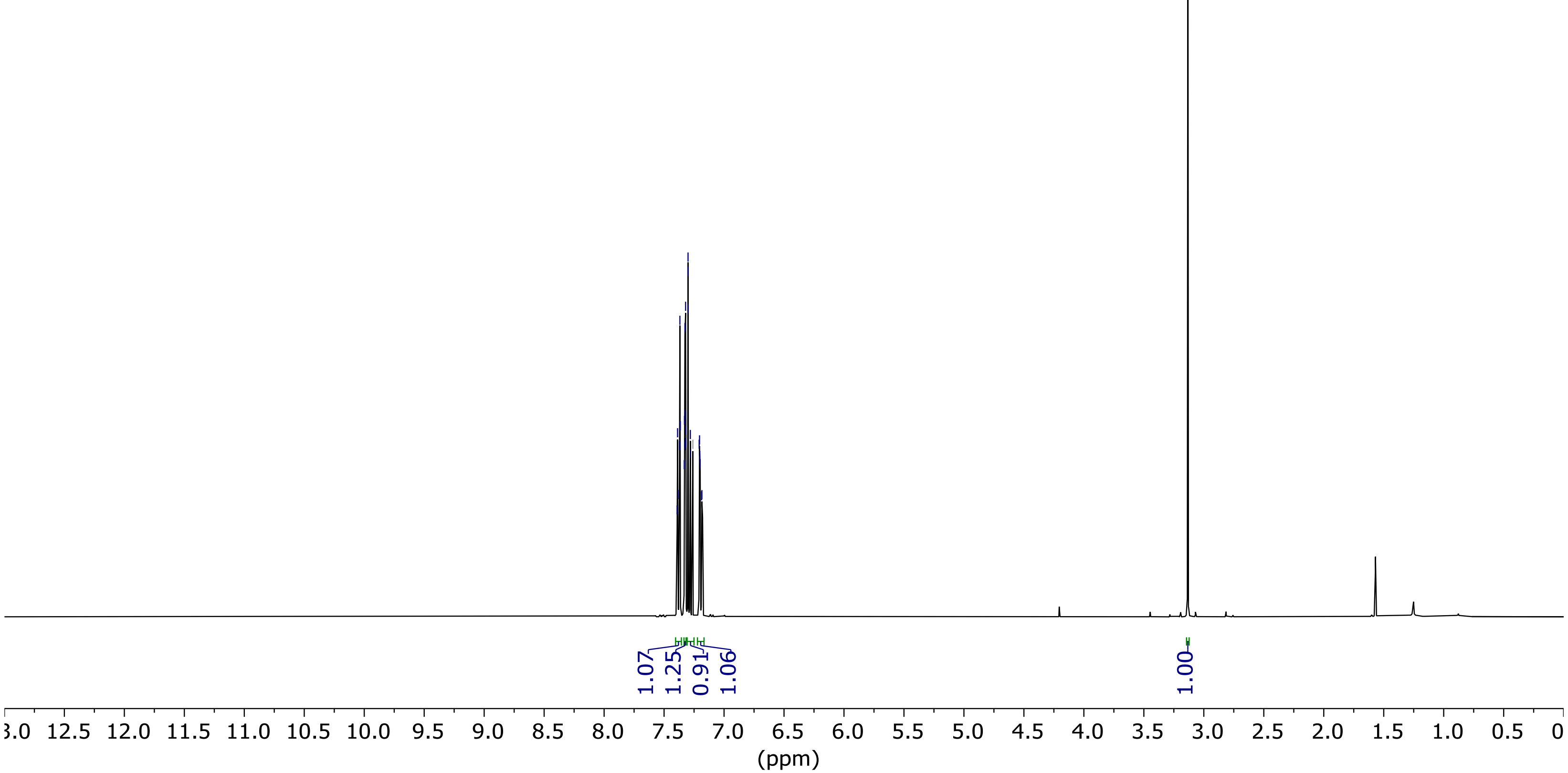
4k

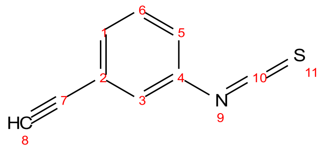




400 MHz, $CDCl_3$

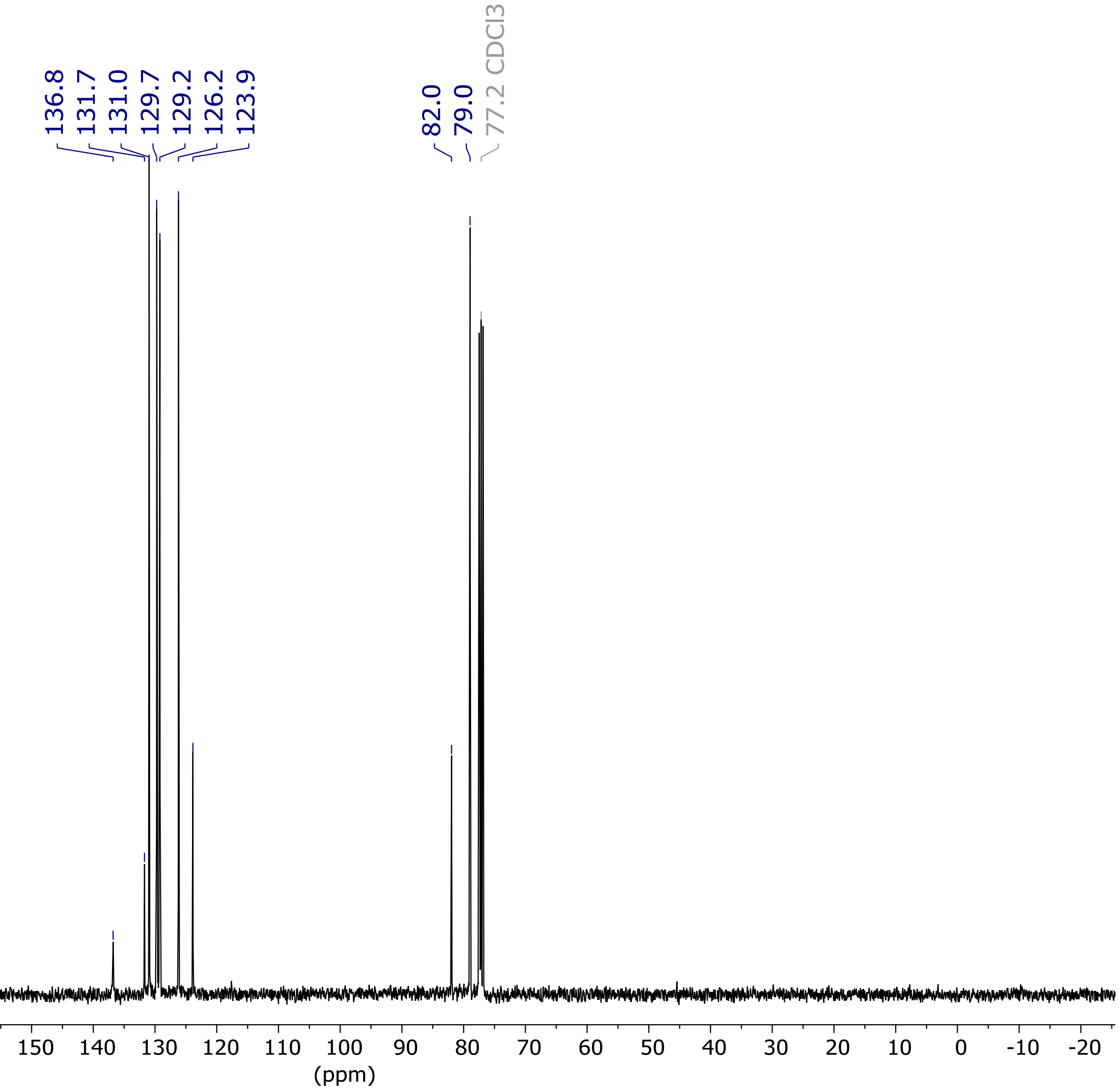
4l

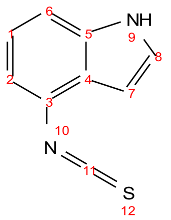




101 MHz, $CDCl_3$

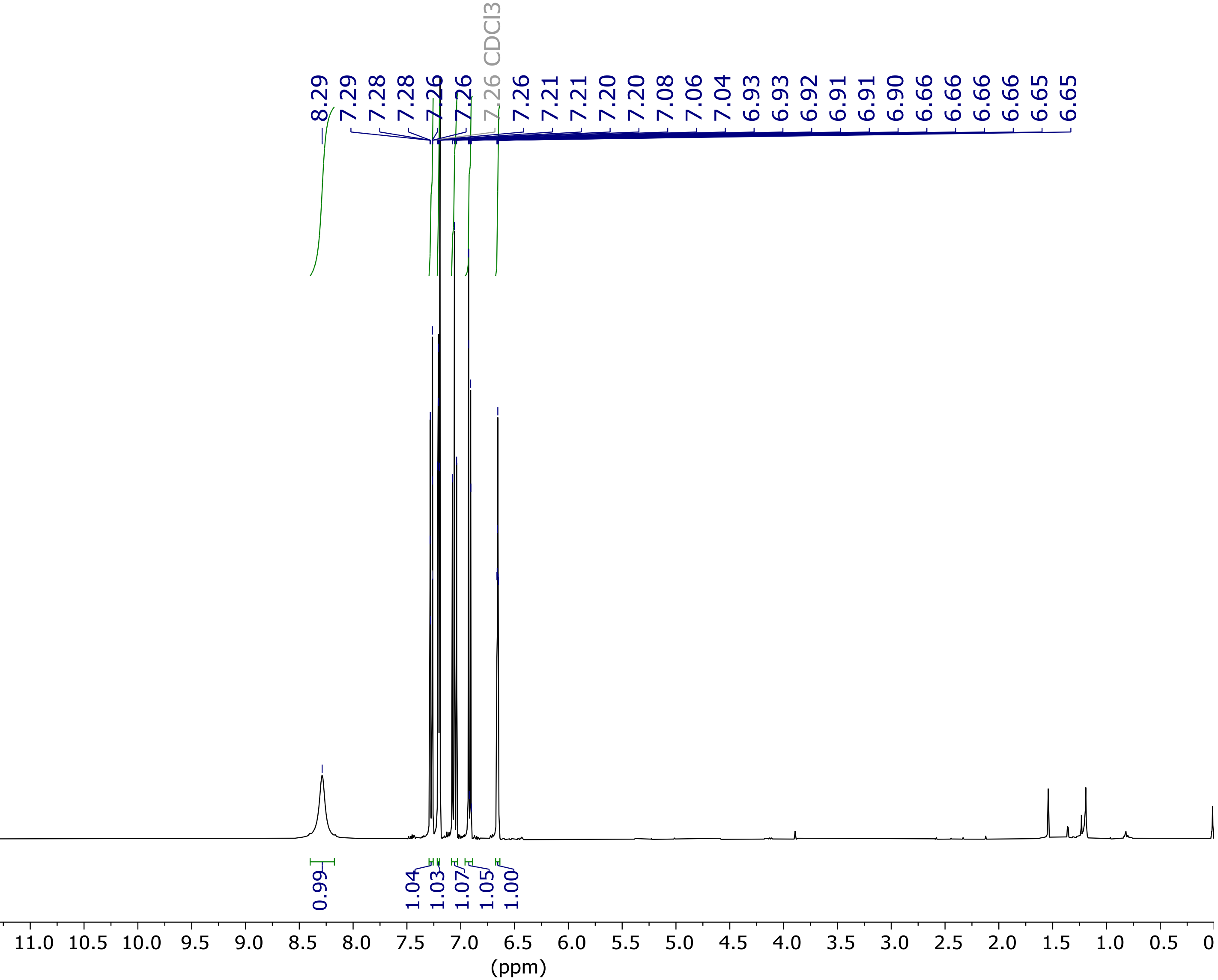
4l

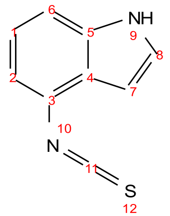




400 MHz, $CDCl_3$

4m





101 MHz, $CDCl_3$

4m