*Harnessing Sulfilimine as an Oxidizing Directing Group in Cp*Co(III)-Catalyzed [4 + 2] Annulation with Alkynes and 1,3-Diynes*

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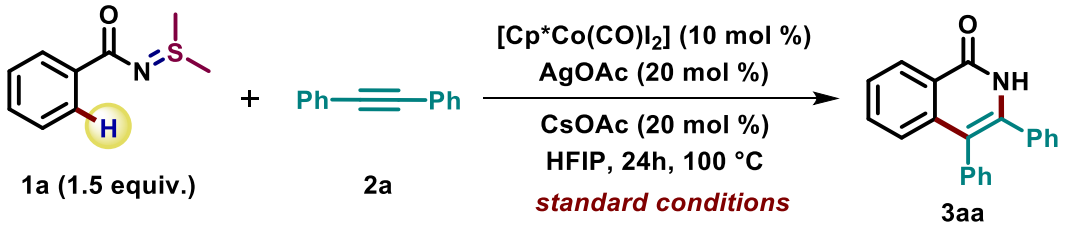
1. General methods

Unless otherwise stated, all commercial reagents and solvents were used without additional purification. Analytical thin layer chromatography (TLC) was performed on precoated silica gel 60 F254 plates. Visualization via TLC was achieved by the use of UV light (254 nm). Column chromatography was undertaken on silica gel (100−200 mesh) using a proper eluent system. NMR spectra were recorded in $CDCl_3$ or DMSO-D_6 at 500 MHz for ^1H NMR spectra, 125 MHz for ^{13}C NMR spectra and 470 MHz for ^{19}F NMR spectra.. Chemical shifts are quoted in parts per million referenced to the appropriate solvent peak or 0.0 ppm for tetramethylsilane. The following abbreviations were used to describe peak splitting patterns when appropriate: br = broad, s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublet, m = multiplet. Coupling constants, *J*, are reported in hertz. For ^{13}C NMR, chemical shifts are reported in parts per million referenced to the center of a triplet at 77.0 ppm of chloroform-d center of a septate at 39.52 ppm in case of DMSO-D_6. HRMS spectra were recorded using ESI-TOF techniques. The sulfilimine were prepared according to the procedure described in the literature.[1] diphenylacetylene is purchased from Alfa Aesar and used without any further purification.

2. Experimental procedure for the optimization study

To a screw-capped vial with a spinvane triangular-shaped Teflon stirbar were added sulfilimine **1a** (27.2 mg, 0.15 mmol, 1.5 equiv.), diphenylacetylene **2a** (17.8 mg, 0.1 mmol, 1.0 equiv.), [Cp*Co(CO)I_2] (4.8 mg, 10.0 mol %), Ag(I) salt (20 mol %), additive (20 mol %), and solvent (0.6 mL) under nitrogen atmosphere. The reaction mixture was stirred at a given temperature in an oil bath for the indicated period of time, filtered through a pad of celite and then the celite pad was washed with CH_2Cl_2 (10 mL × 2). The solvent was removed under reduced pressure and crude yield was measured by ^1H NMR using an internal standard (1,1,2,2-tetrachloroethane). The yields were calculated with respect to diphenylacetylene (limiting agent).

Table S1. Optimization study[a]



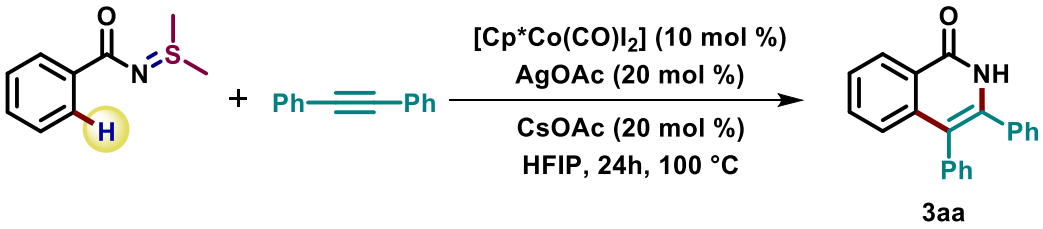
Entry	Variation from standard conditions	Yield (%)[b]
1	none	76 (72)[c]
2	Without AgOAc	trace
3	KOAc instead of CsOAc	72
4	NaOAc instead of CsOAc	68
5	PivOH instead of CsOAc	46
6	AcOH instead of CsOAc	48
7	1,2-DCE as a solvent instead of HFIP	n.d.
8	TFE as a solvent instead of HFIP	65
9	$AgSbF_6$ instead of AgOAc	40
10	$AgNTf_2$ instead of AgOAc	50
11	12 h instead of 24 h	60
12	80 °C instead of 100 °C	58
13	without CsOAc	65
14	without [Cp*Co(CO)I_2]	n.d.

[a]Reaction conditions: **1a** (0.15 mmol, 1.5 equiv.), **2a** (0.10 mmol), Ag(I) salt (20 mol %), additive (20 mol %), and solvent (0.6 mL) at indicated temperature and time. [b]Yields are based on crude ^{1}H NMR (internal standard: 1,1,2,2 tetrachloroethane). [c]Isolated yield n.d. = not detected. HFIP = 1,1,1,3,3,3-Hexafluoro-2-propanol, PivOH = Pivalic acid.

3. General procedure for the Co-catalyzed C–H annulation of sulfilimines with alkynes and 1,3diynes

To a 5 mL seal tube with a Teflon stirbar were added sulfilimine **1** (0.45 mmol, 1.5 equiv.), alkyne/1,3-diyne **2** (0.3 mmol, 1.0 equiv.), [Cp*Co(CO)I_2] (14.3 mg, 10.0 mol %), AgOAc (10.0 mg, 20 mol %), CsOAc (11.5 mg, 20 mol %), and HFIP (1.8 mL) under inert atmosphere. The reaction mixture was stirred at 100 °C in an oil bath for 24 h, filtered through a pad of celite and then the celite pad was washed with CH_2Cl_2 (25 mL × 2). The solvent was removed under reduced pressure and the residue was purified by column chromatography on silica gel using EtOAc/*n*-Hexane) as the eluent to furnish the required isoquinolones.

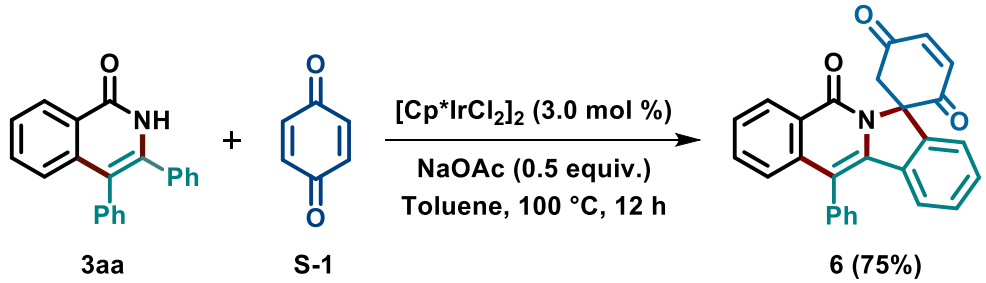
4. Large-scale reaction



To a 10 mL seal tube with a Teflon stirbar were added added sulfilimine **1** (400 mg, 2.2 mmol, 1.5 equiv.), diphenylacetylene **2a** (262 mg, 1.47 mmol, 1.0 equiv.), [Cp*Co(CO)I_2] (70.0 mg, 10.0 mol %), AgOAc (49.0 mg, 20 mol %), CsOAc (56.5 mg, 20 mol %), and HFIP (4.0 mL) under air atmosphere. The reaction mixture was stirred at 100 °C in an oil bath for 24 h, filtered through a pad of celite and then the celite pad was washed with CH_2Cl_2 (25 mL × 2). The solvents were removed under reduced pressure and the residue was purified by column chromatography on silica gel (25 to 30% EtOAc in *n*-Hexane) as the eluent afforded the product (**3aa**) with 68% yield (297.0 mg).

5. Post-synthetic modifications of isoquinolone 3aa

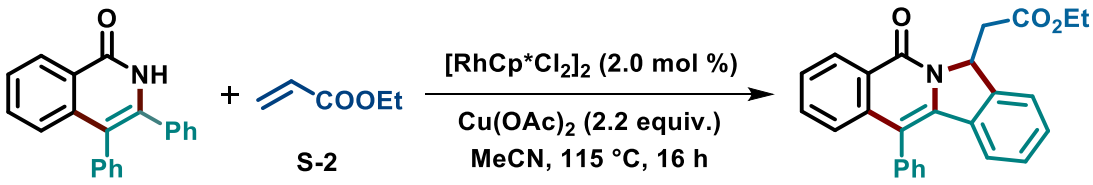
(a) Synthesis of 12'-phenyl-5'*H*-spiro[cyclohexane-1,7'-isoindolo[2,1-*b*]isoquinolin]-3-ene-2,5,5'-trione (6)



To a 5.0 mL seal tube with a Teflon stirbar were added **3aa** (59.4 mg, 0.20 mmol), benzoquinone **S-1** (47.6 mg, 0.44 mmol, 2.2 equiv), [Cp*$IrCl_2$]$_2$ (4.8 mg, 0.006 mmol, 3.0 mol %), and NaOAc (8.2 mg, 0.1 mmol, 0.5 equiv.) and toluene (1.0 mL) under air atmosphere. The reaction mixture was stirred at 100 °C for 12 h. After completion of reaction, the reaction mixture was filtered through a pad of celite and then the celite pad was washed with CH_2Cl_2 (25 mL × 2). The organic layer was removed under reduced pressure and the crude residue was purified by column chromatography on silica gel (15 to 20% of EtOAc in *n*-Hexane) to furnish the product **6** as yellow solid (60.6 mg, 75%).[2] **^{1}H NMR (500 MHz, $CDCl_3$)** δ 8.46 (dd, *J* =

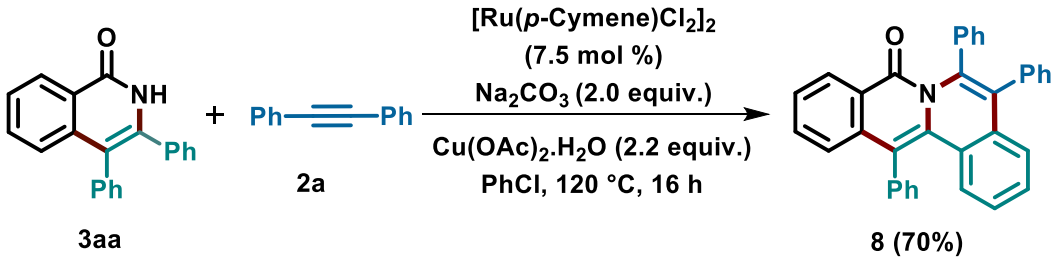
8.2, 1.0 Hz, 1H), 7.65 – 7.59 (m, 4H), 7.52 (t, J = 7.5 Hz, 1H), 7.47 – 7.41 (m, 2H), 7.29 – 7.25 (m, 1H), 7.23 (d, J = 8.2 Hz, 1H), 7.20 (d, J = 7.7 Hz, 1H), 7.17 (s, 2H), 7.15 – 7.11 (m, 1H), 6.41 (d, J = 8.2 Hz, 1H), 4.91 (d, J = 16.0 Hz, 1H), 2.97 (d, J = 16.0 Hz, 1H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 195.2, 189.2, 160.3, 142.8, 141.4, 140.0, 138.9, 136.9, 134.5, 133.2, 132.7, 130.9, 130.8, 129.8, 129.7, 129.6, 129.5, 128.7, 127.5, 126.9, 125.6, 125.1, 125.0, 121.0, 115.3, 74.5, 45.9.

(b) Synthesis of ethyl 2-(5-oxo-12-phenyl-5,7-dihydroisoindolo[2,1-*b*]isoquinolin-7-yl)acetate (7)



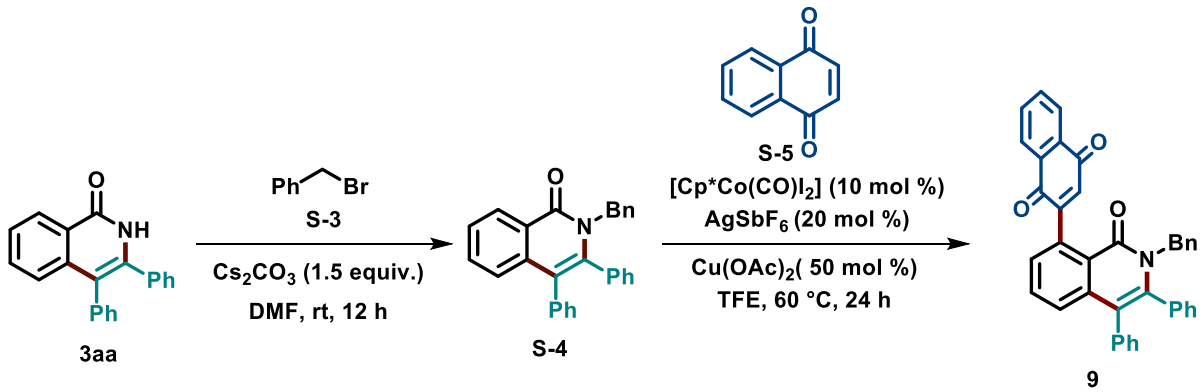
To a 5.0 mL seal tube with a Teflon stirbar were added **3aa** (0.10 mmol, 1.0 equiv.), ethyl acrylate **S-2** (20.2 mg, 2.0 equiv.), [RhCp*Cl_2]$_2$ (1.2 mg, 2 mol %), $Cu(OAc)_2$ (40 mg, 2.2 equiv.), and acetonitrile (0.8 mL). The reaction mixture was stirred at 115 °C in an oil bath for 16 h. After completion of reaction (TLC), the reaction mixture was filtered through a pad of celite and then the celite pad was washed with CH_2Cl_2 (15 mL × 2). The organic layer was removed under reduced pressure and the crude residue was purified by column chromatography on silica gel (30 to 35% of EtOAc in *n*-Hexane) to furnish the product **7** as white solid (24 mg, 60%).[3] **^{1}H NMR (500 MHz, $CDCl_3$)** δ 8.54 (dd, J = 7.7, 1.0 Hz, 1H), 7.63 – 7.43 (m, 7H), 7.37 – 7.30 (m, 2H), 7.20 (d, J = 8.0 Hz, 1H), 7.09 (t, J = 7.7 Hz, 1H), 6.38 (d, J = 7.9 Hz, 1H), 6.01 (dd, J = 7.7, 3.6 Hz, 1H), 4.11 (q, J = 7.1 Hz, 2H), 3.73 (dd, J = 16.1, 3.7 Hz, 1H), 3.06 (dd, J = 16.0, 7.7 Hz, 1H), 1.15 (t, J = 7.1 Hz, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 170.4, 160.9, 141.6, 138.7, 137.9, 135.1, 133.5, 132.1, 131.1, 130.9, 129.5, 129.4, 128.43, 128.38, 127.3, 126.3, 125.2, 124.8, 123.9, 122.8, 114.5, 60.6, 56.0, 36.6, 14.0 (one carbon is missing in the aromatic region due to the overlap).

(c) Synthesis of 5,6,13-triphenyl-8*H*-isoquinolino[3,2-*a*]isoquinolin-8-one (8)



To a 5.0 mL seal tube with a Teflon stirbar were added **3aa** (0.10 mmol, 1.0 equiv.), diphenyl acetylene **2a** (0.20 mmol, 2.0 equiv.), $[RuCl_2(p\text{-cymene})]_2$ (4.6 mg, 7.5 mol %), $Cu(OAc)_2.H_2O$ (43.8 mg, 0.22 mmol, 2.2 equiv.), Na_2CO_3 (21.2 mg, 0.20 mmol, 2.0 equiv.) and PhCl (1.0 mL). The reaction mixture was stirred at 120 °C in an oil bath for 16 h. After completion of reaction (TLC), the reaction mixture was filtered through a pad of celite and then the celite pad was washed with CH_2Cl_2 (15 mL × 2). The organic layer was removed under reduced pressure and the crude residue was purified by column chromatography on silica gel (30 to 35% of EtOAc in *n*-Hexane) to furnish the product **8** as a yellow solid (33.2 mg, 70%).[4] **^{1}H NMR (500 MHz, $CDCl_3$)** δ 8.25 (d, *J* = 8.0 Hz, 1H), 7.62 – 7.57 (m, 1H), 7.56 – 7.49 (m, 5H), 7.43 (t, *J* = 7.5 Hz, 1H), 7.34 (d, *J* = 8.3 Hz, 1H), 7.28 – 7.20 (m, 3H), 7.17 – 7.10 (m, 5H), 7.09 – 7.04 (m, 5H), 6.89 – 6.84 (m, 1H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 162.2, 138.5, 137.1, 136.2, 136.1, 133.7, 133.0, 132.3, 132.1, 131.4, 129.7, 129.0, 128.8, 128.4, 128.1, 127.9, 127.6, 127.4, 127.1, 126.9, 126.8, 126.7, 126.4, 126.3, 125.7, 125.6, 125.5, 116.9 (one carbon is missing in the aromatic region due to the overlap).

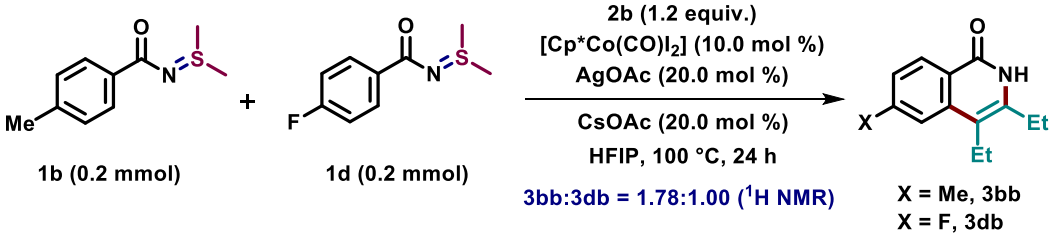
(d) Synthesis of ethyl 2-(2-benzyl-1-oxo-3,4-diphenyl-1,2-dihydroisoquinolin-8-yl)naphthalene-1,4-dione (9)



3,4-diphenylisoquinolin-1(2*H*)-one (**3aa**) (0.33 mmol) were taken in a 10 mL round bottom flask and charged with magnetic stirring bars and dissolved in 1.0 mL DMF. After that Cs_2CO_3 (161.2 mg, 1.5 equiv.) and benzyl bromide **S-3** (86 mg, 1.5 equiv.) were added to it drop wise. The reaction mixture was allowed to stir at room temperature for overnight. After completion of the reaction mixture, water was added to quench the reaction. The aqueous layer was extract with ethyl acetate for three times. The combined organic layer were dried over anhydrous Na_2SO_4 and removed under reduced pressure. The product were purified by silica gel column chromatography using ethyl acetate-hexane mixture (1:3) as the eluent to furnish the product **S-4** as White solid, (65 mg, 50%).[5] **^{1}H NMR (500 MHz, $CDCl_3$)** δ 8.61 (dd, *J* = 8.1, 1.1 Hz, 1H), 7.59 – 7.49 (m, 2H), 7.19 – 7.10 (m, 8H), 7.08 – 7.03 (m, 4H), 6.91 – 6.87 (m, 4H), 5.21 (brs, 2H); **HRMS (ESI)** m/z calcd. for $C_{28}H_{21}NNaO$ $[M+Na]^+$: 410.1521, found: 410.1521.

To a screw-capped vial with a spinvane triangular-shaped Teflon stirbar were added 2-benzyl-3,4-diphenylisoquinolin-1(2*H*)-one **S-4** (0.10 mmol, 1.0 equiv.), naphthoquinone **S-5** (19.0 mg, 1.2 equiv), $[Cp^*Co(CO)I_2]_2$ (4.8 mg, 10 mol %), $AgSbF_6$ (6.8 mg, 20 mol %), $Cu(OAc)_2$ (9.1 mg, 50 mol %), and TFE (0.6 mL). The reaction mixture was stirred at 60 °C in an oil bath for 24 h. Then, the reaction mixture was filtered through a pad of celite and then the celite pad was washed with CH_2Cl_2 (15 mL × 2). The organic layer was removed under reduced pressure and the crude residue was purified by column chromatography on silica gel (10 to 15% of EtOAc in *n*-Hexane) to furnish the product **9** as a brown gummy solid (11 mg, 20%). **^{1}H NMR (500 MHz, $CDCl_3$)** δ 8.17 – 8.13 (m, 1H), 8.12 – 8.08 (m, 1H), 7.75 – 7.69 (m, 2H), 7.65 – 7.60 (m, 1H), 7.44 – 7.39 (m, 1H), 7.31 (dd, *J* = 8.2, 1.1 Hz, 1H), 7.21 – 7.12 (m, 6H), 7.12 – 7.00 (m, 5H), 6.92 (s, 1H), 6.88 – 6.74 (m, 4H), 5.08 (br s, 2H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 185.6, 183.9, 161.9, 155.0, 142.1, 138.4, 137.1, 136.2, 136.0, 133.9, 133.5, 133.4, 133.3, 132.6, 131.9, 131.5, 130.3, 130.2, 128.5, 128.20, 128.12, 128.1, 127.9, 127.6, 127.6, 127.5, 126.94 126.87, 126.8, 126.8, 126.1, 123.9, 119.6, 49.1; **HRMS (ESI)** m/z calcd. for $C_{38}H_{25}NaNO_3$ $[M+Na]^+$: 566.1732, found: 566.1737.

6. Intermolecular competitive experiment of sulfilimines



To a dried screw capped vial with a spinvane triangular-shaped Teflon stirbar were added **1b** (39.1 mg, 0.20 mmol), **1d** (39.9 mg, 0.20 mmol), 3 - hexyne **2b** (19.7 mg, 1.2 equiv.), followed by [Cp*Co(CO)I_2] (9.5 mg, 10 mol %), AgOAc (6.7 mg, 20 mol %), CsOAc (7.7 mg, 20 mol %), and HFIP (1.2 mL) under an inert atmosphere. The reaction mixture was stirred at 100 °C in an oil bath for 24 h. Then, the reaction mixture was filtered through a pad of celite and then the celite pad was washed with CH_2Cl_2 (10 mL × 2). The combined organic layers were removed under reduced pressure. The crude residue was purified by column chromatography on silica gel and ratio of the products **3bb** and **3db** was measured by ^{1}H NMR analysis which was found to be **1.78:1.00.**

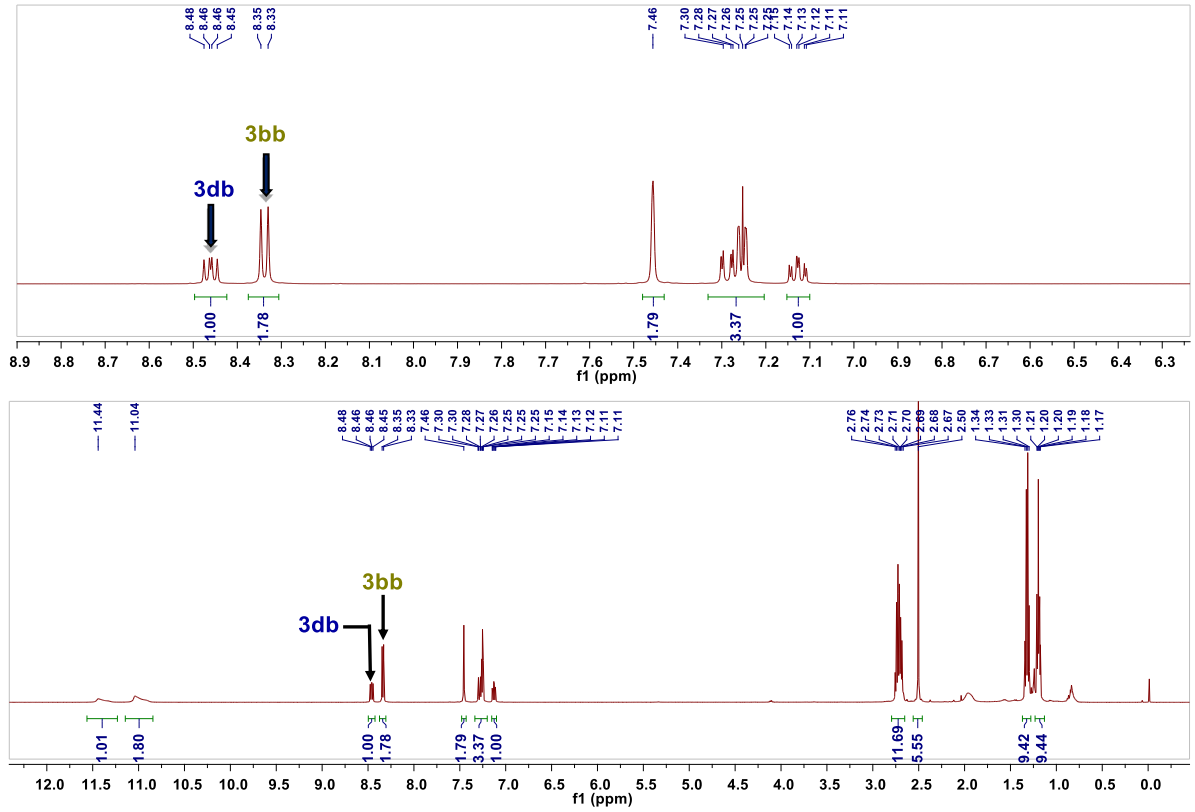
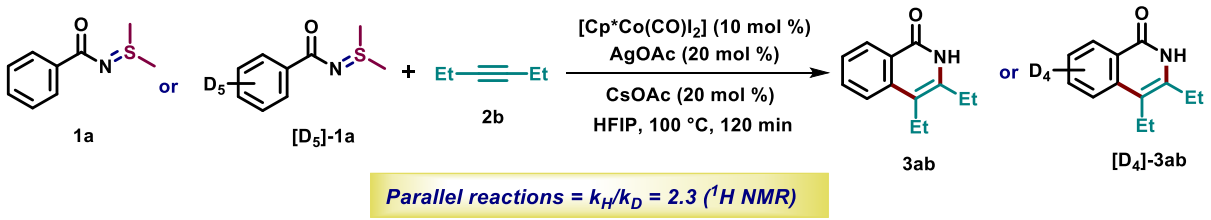


Figure S1. ^{1}H NMR for intermolecular competitive experiment between **1b** and **1d.**

7. Kinetic isotope effect study: Parallel reactions



sulfilimine **1a** (18.1 mg, 0.10 mmol) and sulfilimine-d_5 **[D$_5$]-1a** (18.6 mg, 0.10 mmol) were added to two separate screw capped vials with spinvane triangular-shaped Teflon stirbar followed by 3-hexyne **2b** (9.9 mg, 1.2 equiv.), [Cp*Co(CO)I$_2$] (4.8 mg, 10 mol %), AgOAc (3.3 mg, 20 mol %), CsOAc (3.8 mg, 20 mol %), and HFIP (0.6 mL) under an inert atmosphere. Then, these two reaction mixtures were stirred side-by-side at 100 °C in an oil bath for 120 min. Then, both the reactions were rapidly combined and filtered through a pad of celite and the celite pad was washed with CH_2Cl_2 (5 mL × 2). The solvent was removed under reduced pressure and the crude residue was purified by column chromatography on silica gel (30 to 35% of EtOAc in *n*-Hexane) to furnish the the desired products **3ab** and **[D$_4$]-3ab**. **KIE = 2.3**

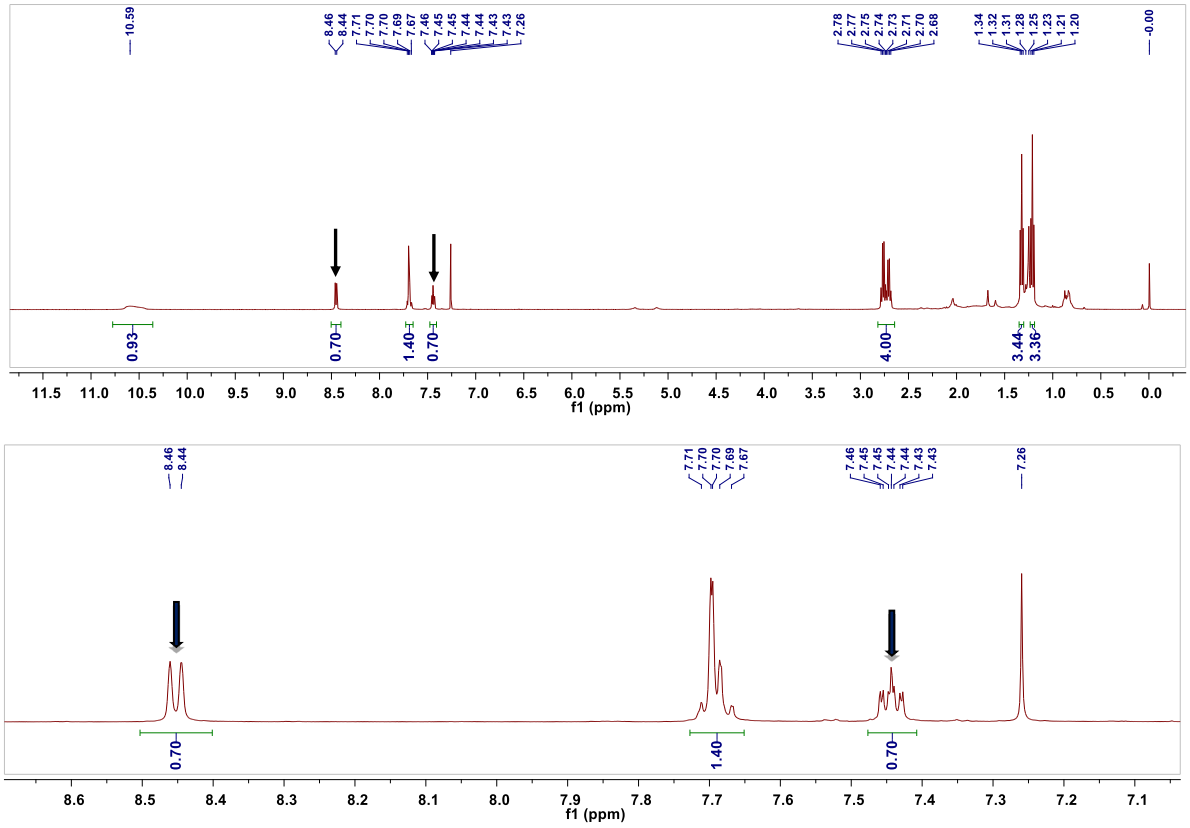
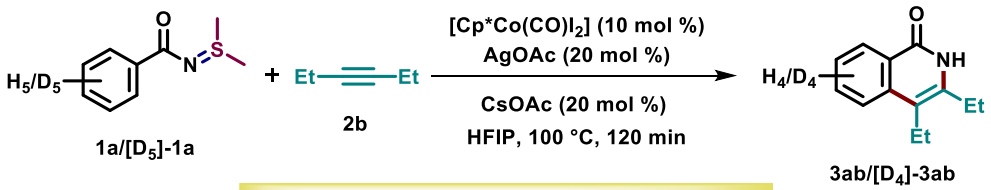


Figure S2. ^{1}H NMR for KIE Study **(Parallel Reactions)**.

8. Kinetic isotope effect study: Competitive reaction



Competitive reaction = k_H/k_D = 3.3 (^{1}H NMR)

To a dried screw capped vial with a spinvane triangular-shaped Teflon stirbar were added **1a** (18.1 mg, 0.10 mmol), **[D_5]-1a** (18.6 mg, 0.10 mmol), 3-hexyne **2b** (9.9 mg, 1.2 equiv.), [Cp*Co(CO)I_2] (10 mol %, 4.8 mg), AgOAc (3.3 mg, 20 mol %), CsOAc (3.8 mg, 20 mol %), and HFIP (0.6 mL) under an Nitrogen atmosphere. The reaction mixture was stirred at 100 °C in an oil bath for 120 min. Then, the reaction mixture was filtered through a pad of celite and then the celite pad was washed with CH_2Cl_2 (5 mL × 2). The combined organic layers were removed under reduced pressure. The crude residue was purified by column chromatography

on silica gel (30 to 35% of EtOAc in *n*-Hexane) to furnish the the desired products **3ab** and **[D_4]-3ab**. **KIE = 3.3**

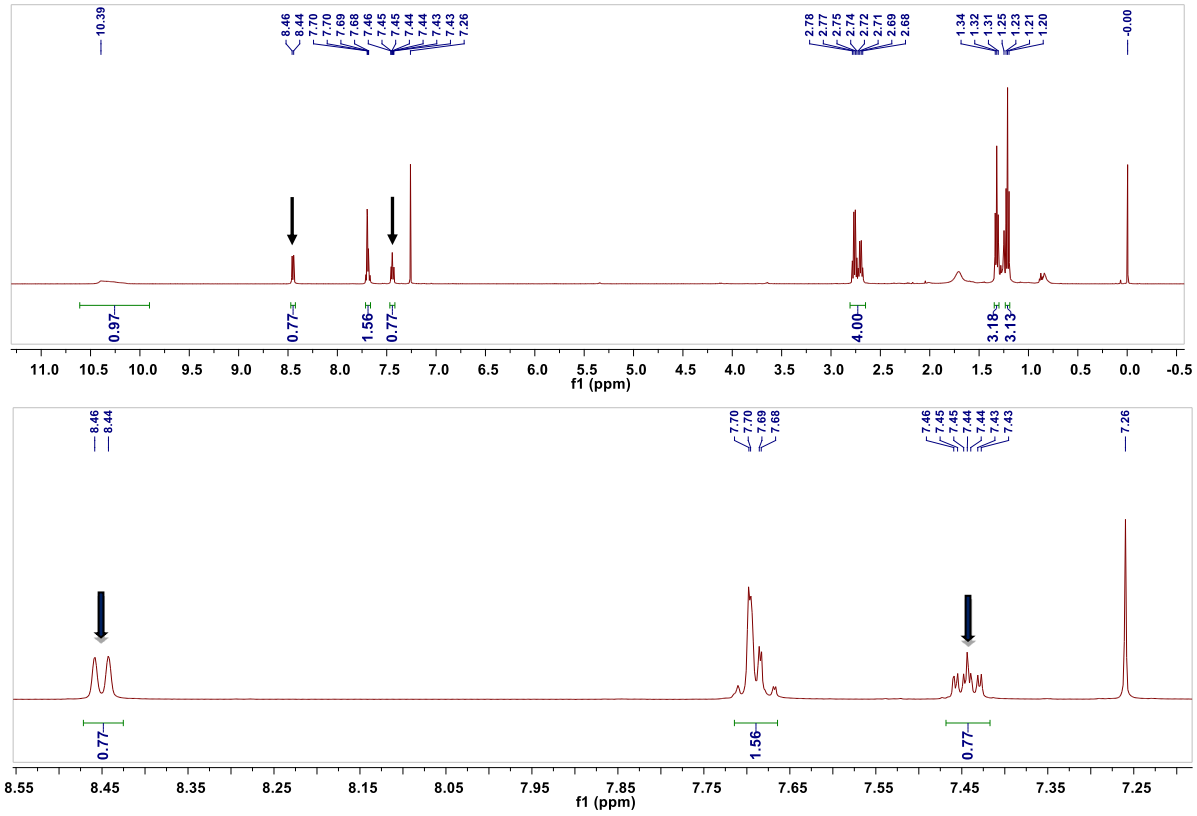
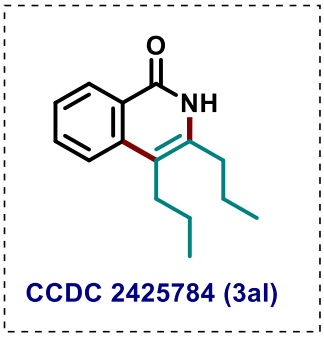


Figure S3. ^{1}H NMR for KIE Study **(Competitive Reaction)**

9. *X-ray structures*

For compound 3al



The purified compound **3al** was dissolved in a solvent of $CHCl_3$, and placed in a dark cabinet for slowly evaporation. Yellow crystals were collected after few days for X-ray analysis.

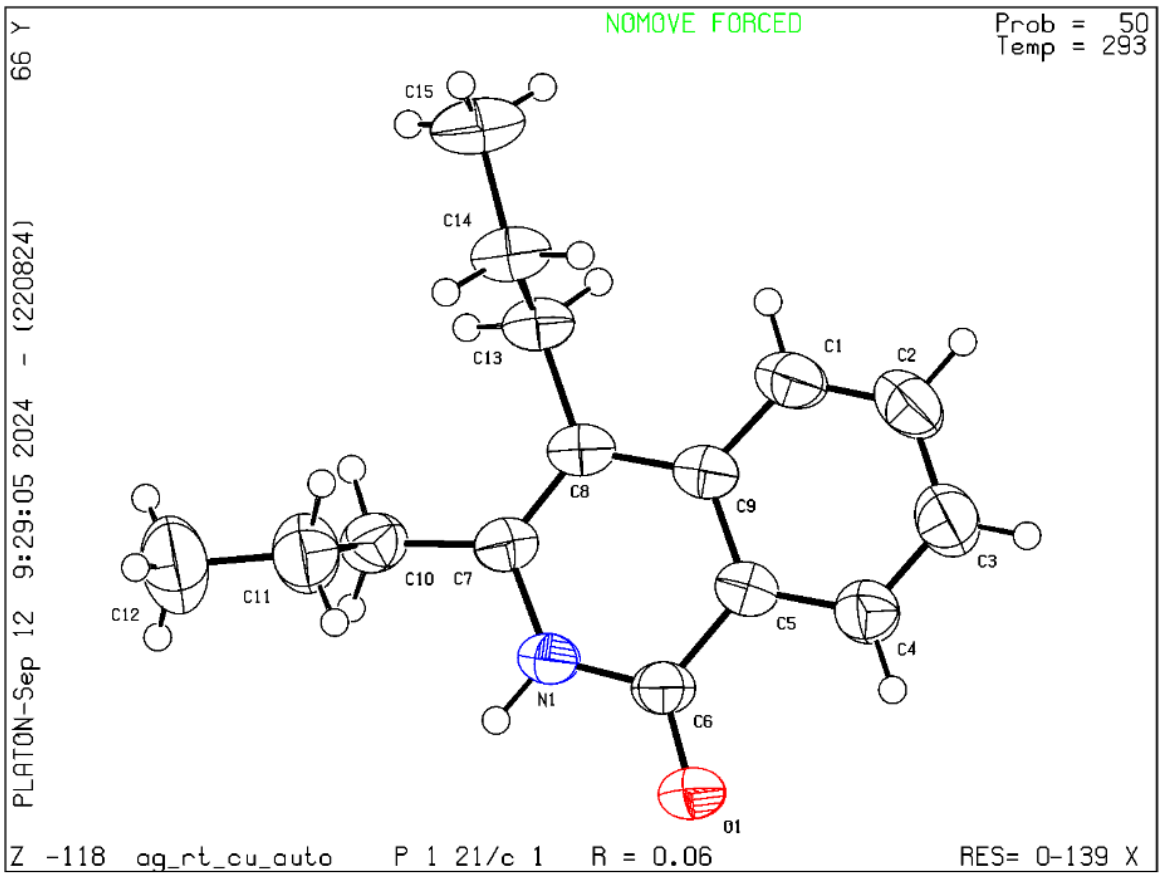


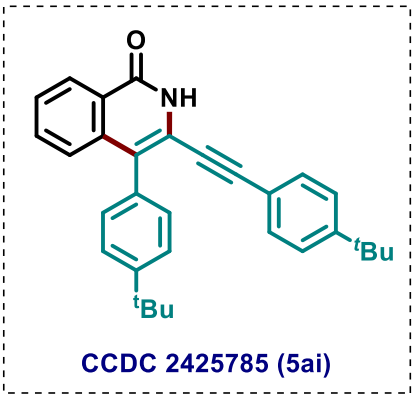
Figure S4: ORTEP diagram of compound **3al** with the atom-numbering. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radius.

Crystal Data Collection and Refinement Parameters for Compound (3al):

Identification code	AG_Rt_Cu_auto
Empirical formula	$C_{15}H_{19}NO$
Formula weight	229.31
Temperature/K	293(2)
Crystal system	Monoclinic
Space group	$P2_1/c$
a/Å	8.7665(7)
b/Å	18.8687(13)
c/Å	8.1487(5)
α/°	90.00

β/°	102.511(7)
γ/°	90.00
Volume/Å^3	1315.89(16)
Z	4
ρ_{calc}g/cm^3	1.157
μ/mm^{-1}	0.559
F(000)	496.0
Crystal size/mm^3	0.642 × 0.152 × 0.144
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	9.374 to 138.302
Index ranges	-10 ≤ h ≤ 10, -15 ≤ k ≤ 22, -9 ≤ l ≤ 9
Reflections collected	3772
Independent reflections	2393 [R_{int} = 0.0205, R_{sigma} = 0.0306]
Data/restraints/parameters	2393/0/160
Goodness-of-fit on F^2	0.987
Final R indexes [I>=2σ (I)]	R_1 = 0.0568, wR_2 = 0.1546
Final R indexes [all data]	R_1 = 0.0721, wR_2 = 0.1760
Largest diff. peak/hole / e Å^{-3}	0.19/-0.35

For compound 5ai



The purified compound **5ai** was dissolved in a solvent of DCM, and placed in a dark cabinet for slowly evaporation. Yellow crystals were collected after few days for X-ray analysis.

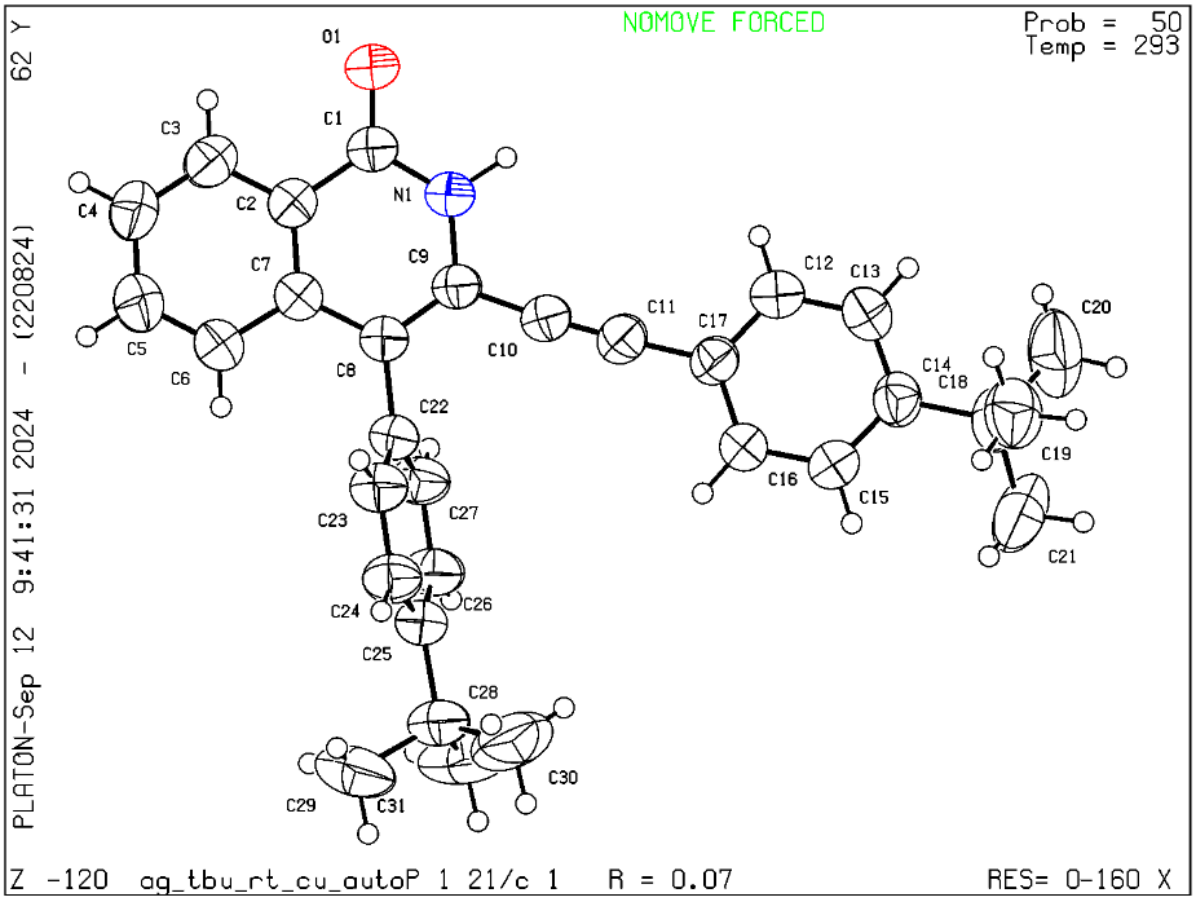


Figure S5: ORTEP diagram of compound **5ai** with the atom-numbering. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radius.

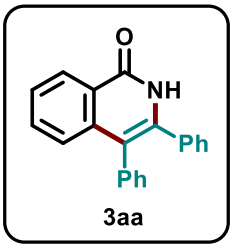
Crystal Data Collection and Refinement Parameters for Compound (5ai):

Identification code	Ag_TBU_Rt_CU_auto
Empirical formula	$C_{31}H_{31}NO$
Formula weight	433.57
Temperature/K	293(2)
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	10.5297(4)
b/Å	13.4796(4)
c/Å	18.0553(5)

α/°	90
β/°	92.422(3)
γ/°	90
Volume/Å^3	2560.41(14)
Z	4
ρ_{calc}g/cm^3	1.125
μ/mm^{-1}	0.513
F(000)	928.0
Crystal size/mm^3	0.385 × 0.303 × 0.179
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	8.188 to 138.138
Index ranges	-12 ≤ h ≤ 12, -7 ≤ k ≤ 16, -21 ≤ l ≤ 20
Reflections collected	7700
Independent reflections	4678 [R_{int} = 0.0309, R_{sigma} = 0.0454]
Data/restraints/parameters	4678/0/308
Goodness-of-fit on F^2	1.030
Final R indexes [I>=2σ (I)]	R_1 = 0.0748, wR_2 = 0.2116
Final R indexes [all data]	R_1 = 0.0953, wR_2 = 0.2507
Largest diff. peak/hole / e Å^{-3}	0.23/-0.33

10. Spectroscopic data of isoquinolones obtained in this study

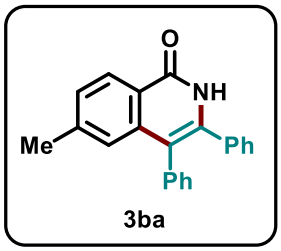
3,4-Diphenylisoquinolin-1(2*H*)-one(3aa).[6]



3aa

Yellow solid (64.4 mg, 72%); eluent (25-30% ethyl acetate in hexane); **^{1}H NMR (500 MHz, DMSO-D$_6$)** δ 11.57 (s, 1H), 8.34 – 8.30 (dd, *J* = 8.0, 1.0 Hz 1H), 7.64 (m, 1H), 7.52 (t, *J* = 7.6 Hz, 1H), 7.32 – 7.25 (m, 3H), 7.24 – 7.21 (m, 5H), 7.15 (dd, *J* = 8.0, 1.1 Hz, 3H). **^{13}C NMR (125 MHz, DMSO-D$_6$)** δ 161.7, 138.6, 138.1, 135.9, 134.6, 132.5, 131.7, 129.9, 128.3, 128.2, 127.7, 127.1, 126.8, 126.3, 125.02, 125.0, 115.5.

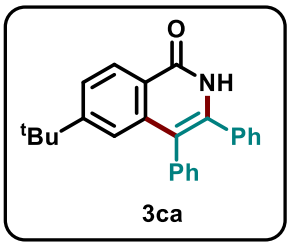
6-Methyl-3,4-diphenylisoquinolin-1(2*H*)-one(3ba).[7]



3ba

Yellow solid (62.7 mg, 67%); eluent (25-30% ethyl acetate in hexane); **^{1}H NMR (500 MHz, DMSO-D$_6$)** δ 11.47 (brs, 1H), 8.21 (d, *J* = 8.1 Hz, 1H), 7.34 (d, *J* = 8.8 Hz, 1H), 7.32 – 7.24 (m, 3H), 7.23 – 7.18 (m, 5H), 7.15 – 7.12 (m, 2H), 6.92 (s, 1H), 2.30 (s, 3H). **^{13}C NMR (125 MHz, DMSO-D$_6$)** δ 161.5, 142.3, 138.6, 138.2, 135.7, 134.6, 131.7, 129.7, 128.1, 128.0, 127.6, 127.5, 126.9, 126.8, 124.4, 122.9, 115.2, 21.5.

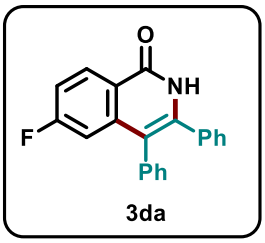
6-(*tert*-Butyl)-3,4-diphenylisoquinolin-1(2*H*)-one(3ca).[1]



3ca

Yellow solid (58.4 mg, 55%); eluent (25-30% ethyl acetate in hexane); **^{1}H NMR (500 MHz, CDCl$_3$)** δ 8.95 (brs, 1H), 8.42 (d, *J* = 8.4 Hz, 1H), 7.57 (dd, *J* = 8.4, 1.9 Hz, 1H), 7.35 (d, *J* = 1.7 Hz, 1H), 7.33 – 7.27 (m, 3H), 7.25 – 7.21 (m, 5H), 7.20 – 7.17 (m, 2H), 1.25 (s, 9H). **^{13}C NMR (125 MHz, CDCl$_3$)** δ 162.6, 156.2, 138.6, 137.0, 135.9, 135.4, 131.9, 129.2, 128.5, 128.3, 128.3, 127.3, 127.2, 124.7, 123.0, 121.8, 117.6, 35.3, 31.0.

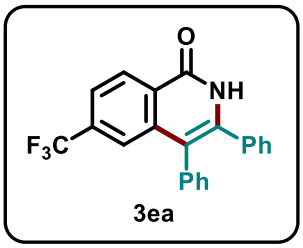
6-Fluoro-3,4-diphenylisoquinolin-1(*2H*)-one(3da).[6]



3da

Yellow solid (56.8 mg, 60%); eluent (25-30% ethyl acetate in hexane); **^{1}H NMR (500 MHz, CDCl$_3$)** δ 10.25 (brs, 1H), 8.42 (dd, *J* = 8.9, 6.0 Hz, 1H), 7.35 – 7.29 (m, 3H), 7.29 – 7.24 (m, 5H), 7.20 – 7.15 (m, 3H), 6.98 (dd, *J* = 10.7, 2.5 Hz, 1H). **^{13}C NMR (125 MHz, CDCl$_3$)** δ 165.5 (d, $J_{C\text{-}F}$ = 250.5 Hz), 162.4, 141.2 (d, $J_{C\text{-}F}$ = 10 Hz), 138.7, 135.3, 134.5, 131.6, 130.6 (d, $J_{C\text{-}F}$ = 5.4 Hz), 129.4, 128.8, 128.5, 128.2, 127.5, 121.6, 116.7 (d, $J_{C\text{-}F}$ = 2.1 Hz), 115.1 (d, $J_{C\text{-}F}$ = 23.5 Hz), 110.8 (d, $J_{C\text{-}F}$ = 23.3 Hz). **^{13}F NMR (470 MHz, CDCl$_3$)** δ –105.0.

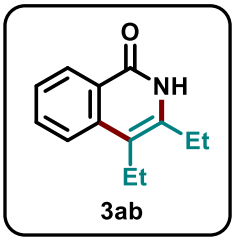
3,4-Diphenyl-6-(trifluoromethyl)isoquinolin-1(2*H*)-one(3ea).[7]



3ea

Yellow solid (51.6 mg, 47%); eluent (25-30% ethyl acetate in hexane); **^{1}H NMR (500 MHz, DMSO-D$_6$)** δ 11.92 (brs, 1H), 8.51 (d, *J* = 8.3 Hz, 1H), 7.81 (dd, *J* = 8.4, 1.3 Hz, 1H), 7.39 (s, 1H), 7.35 – 7.28 (m, 3H), 7.27 – 7.22 (m, 5H), 7.18 (dd, *J* = 7.7, 1.4 Hz, 2H); **^{13}C NMR (125 MHz, DMSO-D$_6$)** δ 161.0, 140.6, 138.3, 134.9, 134.1, 132.2 (q, $J_{C\text{-}F}$ = 31.8 Hz), 131.6, 129.8, 128.6, 128.5, 127.7, 127.5, 127.4, 123.7 (q, $J_{C\text{-}F}$ = 272.9 Hz), 121.9 (d, $J_{C\text{-}F}$ = 2.7 Hz), 121.6 (d, $J_{C\text{-}F}$ = 3.2 Hz), 115.1; **^{13}F NMR (470 MHz, CDCl$_3$)** δ –61.8.

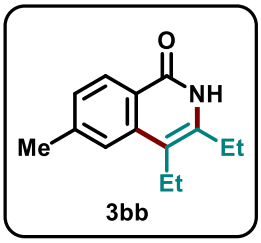
3,4-Diethylisoquinolin-1(2*H*)-one (3ab).[1]



3ab

Yellow solid (40.6 mg, 67%); eluent (30-35% ethyl acetate in hexane); **^{1}H NMR (500 MHz, $CDCl_3$)** δ 11.71 (brs, 1H), 8.49 (d, *J* = 8.0 Hz, 1H), 7.68 (m, 2H), 7.46 – 7.42 (m, 1H), 2.80 – 2.72 (m, 4H), 1.35 (t, *J* = 7.6 Hz, 3H), 1.21 (t, *J* = 7.5 Hz, 3H). **^{13}C NMR (125 MHz, $CDCl_3$)** δ 164.1, 139.4, 138.2, 132.3, 127.7, 125.2, 125.0, 122.8, 113.8, 24.2, 19.5, 14.9, 14.1.

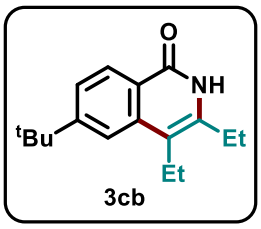
3,4-Diethyl-6-methylisoquinolin-1(2*H*)-one (3bb).



3bb

Yellow solid (41 mg, 63%); eluent (30-35% ethyl acetate in hexane); m.p. 178 – 180 °C; **^{1}H NMR (500 MHz, $CDCl_3$)** δ 11.31 (brs, 1H), 8.36 (d, *J* = 8.2 Hz, 1H), 7.46 (s, 1H), 7.26 (d, *J* = 8.1 Hz, 1H), 2.78 – 2.68 (m, 4H), 2.51 (s, 3H), 1.33 (t, *J* = 7.6 Hz, 3H), 1.21 (t, *J* = 7.5 Hz, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 163.9, 142.7, 139.3, 138.3, 127.7, 126.8, 122.8, 122.6, 113.6, 24.2, 22.3, 19.4, 14.9, 14.0; **HRMS (ESI)** m/z calcd. for $C_{14}H_{18}NO$ $[M+H]^+$: 216.1388, found: 216.1386.

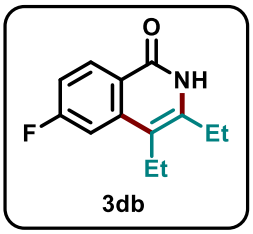
6-(*tert*-Butyl)-3,4-diethylisoquinolin-1(2*H*)-one (3cb).



3cb

Yellow solid (46.4 mg, 60%); eluent (30-35% ethyl acetate in hexane); m.p. 260 – 262 °C; **^{1}H NMR (500 MHz, $CDCl_3$)** δ 11.11 (brs, 1H), 8.39 (d, *J* = 8.4 Hz, 1H), 7.67 (d, *J* = 1.6 Hz, 1H), 7.51 (dd, *J* = 8.4, 1.8 Hz, 1H), 2.78 (q, *J* = 7.5 Hz, 2H), 2.72 (q, *J* = 7.6 Hz, 2H), 1.41 (s, 9H), 1.31 (t, *J* = 7.6 Hz, 3H), 1.23 (t, *J* = 7.5 Hz, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 163.7, 155.6, 139.0, 138.0, 127.5, 123.4, 122.8, 118.7, 114.0, 35.3, 31.2, 24.3, 19.5, 14.9, 14.1; **HRMS (ESI)** m/z calcd. for $C_{17}H_{24}NO$ $[M+H]^+$: 258.1858, found: 258.1854.

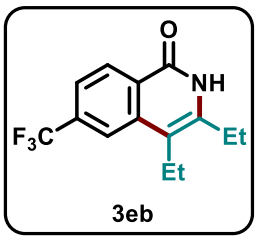
3,4-Diethyl-6-fluoroisoquinolin-1(2*H*)-one (3db).



3db

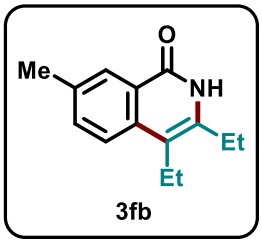
Yellow solid (37.5 mg, 57%); eluent (30-35% ethyl acetate in hexane); m.p. 215 – 220 °C; **^{1}H NMR (500 MHz, $CDCl_3$)** δ 11.42 (brs, 1H), 8.47 (dd, *J* = 8.9, 6.2 Hz, 1H), 7.30 (dd, *J* = 11.0, 2.5 Hz, 1H), 7.14 (td, *J* = 8.5, 2.3 Hz, 1H), 2.77 – 2.67 (m, 4H), 1.34 (t, *J* = 7.6 Hz, 3H), 1.20 (t, *J* = 7.5 Hz, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 166.8 (d, $J_{C\text{-}F}$ = 249.8 Hz), 163.2, 140.9 (d, $J_{C\text{-}F}$ = 9.6 Hz), 140.7, 130.9 (d, $J_{C\text{-}F}$ = 10.3 Hz), 121.9, 113.9 (d, $J_{C\text{-}F}$ = 23.5 Hz), 113.5 (d, $J_{C\text{-}F}$ = 3 Hz), 108.1 (d, $J_{C\text{-}F}$ = 22.5 Hz), 24.4, 19.7, 14.7, 13.8; **HRMS (ESI)** m/z calcd. for $C_{13}H_{15}NOF$ $[M+H]^+$: 220.1138, found: 220.1137.

3,4-Diethyl-6-(trifluoromethyl)isoquinolin-1(2*H*)-one (3eb).



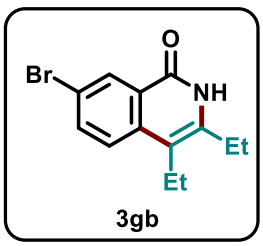
Yellow solid (32.3 mg, 40%); eluent (30-35% ethyl acetate in hexane); m.p. 218 – 220 °C; **^{1}H NMR (500 MHz, $CDCl_3$)** δ 11.55 (brs, 1H), 8.57 (d, *J* = 8.3 Hz, 1H), 7.95 (s, 1H), 7.64 (dd, *J* = 8.3, 1.0 Hz, 1H), 2.83 – 2.78 – 2.70 (m, 4H), 1.36 (t, *J* = 7.6 Hz, 3H), 1.23 (t, *J* = 7.5 Hz, 3H); **^{13}C NMR (125 MHz, DMSO-D_6)** δ 161.0, 142.1, 137.8, 132.3 (q, *J* = 31.3 Hz), 128.7, 127.5, 124.0 (q, *J* = 272.9 Hz), 120.8 (d, *J* = 1.9 Hz), 119.9 (d, *J* = 3.5 Hz), 111.8, 23.3, 18.6, 14.8, 14.0; **^{13}F NMR (470 MHz, $CDCl_3$)** δ –62.8. **HRMS (ESI)** m/z calcd. for $C_{14}H_{14}NNaOF_3$ $[M+Na]^+$: 292.0925, found: 292.0925.

3,4-Diethyl-7-methylisoquinolin-1(2*H*)-one (3fb).



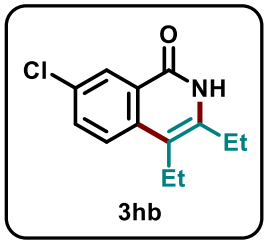
White solid (32 mg, 49%); eluent (30-35% ethyl acetate in hexane); m.p. 163 – 165 °C; **^{1}H NMR (500 MHz, $CDCl_3$)** δ 11.38 (brs, 1H), 8.27 (s, 1H), 7.60 (d, *J* = 8.3 Hz, 1H), 7.50 (dd, *J* = 8.4, 1.8 Hz, 1H), 2.74 (m, 4H), 2.49 (s, 3H), 1.34 (t, *J* = 7.6 Hz, 3H), 1.20 (t, *J* = 7.5 Hz, 3H);**^{13}C NMR (125 MHz, $CDCl_3$)** δ 163.9, 138.2, 135.9, 135.1, 133.9, 127.2, 125.0, 122.8, 113.8, 24.1, 21.1, 19.5, 15.0, 14.1; **HRMS (ESI)** m/z calcd. for $C_{14}H_{18}NO$ $[M+H]^+$: 216.1388, found: 216.1386.

7-Bromo-3,4-diethylisoquinolin-1(2*H*)-one (3gb).



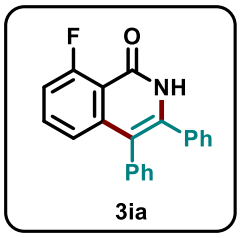
Yellow solid (35.4 mg, 42%); eluent (30-35% ethyl acetate in hexane); m.p. 190 – 192 °C; **^{1}H NMR (500 MHz, $CDCl_3$)** δ 11.61 (brs, 1H), 8.58 (d, *J* = 2.2 Hz, 1H), 7.75 (dd, *J* = 8.8, 2.2 Hz, 1H), 7.56 (d, *J* = 8.7 Hz, 1H), 2.78 – 2.68 (m, 4H), 1.35 (t, *J* = 7.6 Hz, 3H), 1.19 (t, *J* = 7.5 Hz, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 162.9, 140.0, 137.0, 135.4, 130.2, 126.5, 124.8, 119.1, 113.7, 24.2, 19.5, 14.9, 14.0; **HRMS (ESI)** m/z calcd. for $C_{13}H_{14}BrNaNO$ $[M+Na]^+$: 302.0156, found: 302.0157.

7-Chloro-3,4-diethylisoquinolin-1(2*H*)-one (3hb).



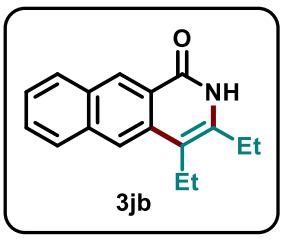
White solid (29.7 mg, 42%); eluent (30-35% ethyl acetate in hexane); m.p. 200 – 202 °C; **^{1}H NMR (500 MHz, $CDCl_3$)** δ 11.30 (brs, 1H), 8.42 (d, *J* = 2.0 Hz, 1H), 7.66 – 7.59 (m, 2H), 2.78 – 2.68 (m, 4H), 1.34 (t, *J* = 7.6 Hz, 3H), 1.20 (t, *J* = 7.5 Hz, 3H). **^{13}C NMR (125 MHz, $CDCl_3$)** δ 162.7, 139.4, 136.7, 132.8, 131.5, 127.2, 126.5, 124.7, 113.6, 24.3, 19.6, 14.8, 13.8; **HRMS (ESI)** m/z calcd. for $C_{13}H_{15}ClNO$ $[M+H]^+$: 236.0842, found: 236.0845.

8-Fluoro-3,4-diphenylisoquinolin-1(2*H*)-one(3ia).[6]



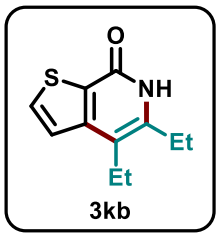
Yellow solid (21 mg, 22%); eluent (25-30% ethyl acetate in hexane); **^{1}H NMR (500 MHz, DMSO-D6)** δ 11.56 (brs, 1H), 7.63 – 7.57 (m, 1H), 7.32 – 7.19 (m, 9H), 7.16 – 7.11 (m, 2H), 6.90 (d, J = 8.2 Hz, 1H). **^{13}C NMR (125 MHz, DMSO-D6)** δ 162.2 (d, $J_{C\text{-}F}$ = 259.9 Hz), 159.7, 141.5, 140.2, 136.0, 134.4, 134.0 (d, $J_{C\text{-}F}$ = 9.9 Hz), 132.0, 130.0, 128.8, 128.7, 128.1, 127.7, 121.4 (d, $J_{C\text{-}F}$ = 2.6 Hz), 115.4, 114.1 (d, $J_{C\text{-}F}$ = 4 Hz), 113.3 (d, $J_{C\text{-}F}$ = 20.6 Hz).**^{13}F NMR (470 MHz, DMSO-D6)** δ –111.0.

3,4-Diethylbenzo[g]isoquinolin-1(2*H*)-one (3jb).



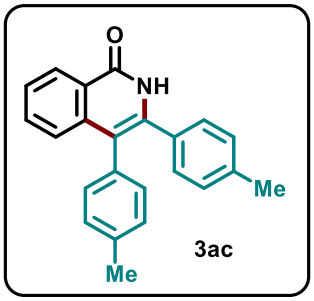
Yellow solid (28.7 mg, 38%); eluent (25-30% ethyl acetate in hexane); m.p. 225 – 227 °C; **^{1}H NMR (500 MHz, CDCl$_3$)** δ 10.63 (brs, 1H), 9.09 (s, 1H), 8.12 (s, 1H), 8.06 (d, J = 8.3 Hz, 1H), 7.96 (d, J = 8.3 Hz, 1H), 7.57 (t, J = 7.6 Hz, 1H), 7.49 (t, J = 7.5 Hz, 1H), 2.88 (q, J = 7.5 Hz, 2H), 2.77 (q, J = 7.6 Hz, 2H), 1.40 (t, J = 7.6 Hz, 3H), 1.30 (t, J = 7.5 Hz, 3H).**^{13}C NMR (125 MHz, CDCl$_3$)** δ 164.4, 137.4, 135.5, 134.1, 130.7, 129.2, 128.8, 127.90, 127.87, 125.5, 124.0, 121.1, 113.4, 24.4, 19.8, 14.8, 13.9; **HRMS (ESI)** m/z calcd. for $C_{17}H_{18}NO$ $[M+H]^+$: 252.1388, found: 252.1388.

4,5-Diethylthieno[2,3-*c*]pyridin-7(6*H*)-one (3kb).



Yellow solid (31.5 mg, 50%); eluent (30-35% ethyl acetate in hexane); m.p. 190 – 192 °C; **^{1}H NMR (500 MHz, CDCl$_3$)** δ 11.92 (brs, 1H), 7.71 (d, J = 5.1 Hz, 1H), 7.28 (d, J = 5.1 Hz, 1H), 2.77 – 2.68 (m, 4H), 1.32 (t, J = 7.6 Hz, 3H), 1.19 (t, J = 7.6 Hz, 3H); **^{13}C NMR (125 MHz, CDCl$_3$)** δ 160.3, 148.1, 141.1, 133.1, 127.3, 122.8, 114.5, 23.5, 21.3, 15.3, 14.5; **HRMS (ESI)** m/z calcd. for $C_{11}H_{13}NaNOS$ $[M+Na]^+$: 230.0616, found: 230.0612.

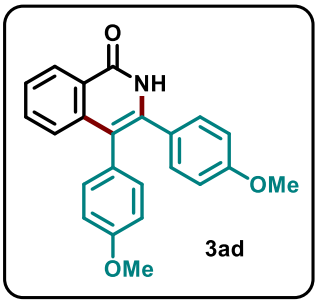
3,4-Di-p-tolylisoquinolin-1(2*H*)-one (3ac).[8]



White solid (62.5 mg, 64%); eluent (25-30% ethyl acetate in hexane); **^{1}H NMR (500 MHz, CDCl$_3$)** δ 9.10 (brs, 1H), 8.48 (d, J = 8.0 Hz, 1H), 7.61 – 7.56 (m, 1H), 7.49 (t, J = 7.5 Hz, 1H), 7.36 (d, J = 8.2 Hz, 1H), 7.15 – 7.10 (m, 4H), 7.08 – 7.03 (m, 4H), 2.36 (s, 3H), 2.30 (s, 3H); **^{13}C NMR (125 MHz, CDCl$_3$)** δ 162.9, 138.9, 138.4, 137.1, 136.8, 132.8, 132.5, 132.1, 131.6, 129.1, 129.1, 128.9,

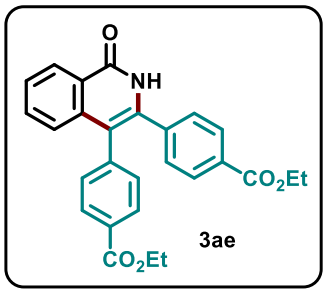
127.3, 126.3, 125.6, 124.9, 116.9, 21.2; (one carbon is missing in the aliphatic region due to the overlap).

3,4-Di-p-tolylisoquinolin-1(2*H*)-one (3ad).[9]



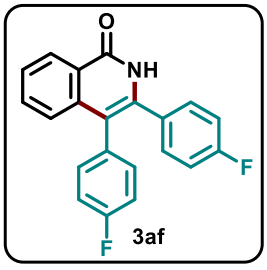
Yellow solid (64.4 mg, 60%); eluent (30-35% ethyl acetate in hexane); m.p. 152 – 154 °C; **^{1}H NMR (500 MHz, $CDCl_3$)** δ 9.58 (brs, 1H), 8.45 (d, *J* = 7.6 Hz, 1H), 7.59 – 7.55 (m, 1H), 7.47 (t, *J* = 7.4 Hz, 1H), 7.36 (d, *J* = 8.2 Hz, 1H), 7.17 (d, *J* = 8.7 Hz, 2H), 7.09 (d, *J* = 8.6 Hz, 2H), 6.86 (d, *J* = 8.5 Hz, 2H), 6.77 (d, *J* = 8.8 Hz, 2H), 3.82 (s, 3H), 3.78 (s, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 162.7, 159.7, 158.8, 139.2, 137.0, 132.9, 132.5, 130.5, 128.2, 127.7, 127.5, 126.3, 125.6, 125.1, 116.4, 114.0, 113.9, 55.2; (one carbon is missing in the aliphatic region due to the overlap).

Diethyl 4,4'-(1-oxo-1,2-dihydroisoquinoline-3,4-diyl)dibenzoate (3ae).[10]



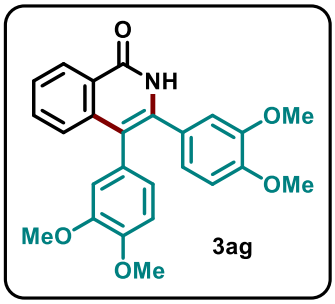
Yellow solid (57.0 mg, 43%); eluent (25-30% ethyl acetate in hexane); **^{1}H NMR (500 MHz, $CDCl_3$)** δ 10.59 (brs, 1H), 8.42 (dd, *J* = 7.8, 1.3 Hz, 1H), 8.00 (d, *J* = 8.2 Hz, 2H), 7.93 (d, *J* = 8.5 Hz, 2H), 7.63 – 7.57 (m, 1H), 7.53 (t, *J* = 7.5 Hz, 1H), 7.33 (d, *J* = 8.4 Hz, 2H), 7.30 (d, *J* = 8.1 Hz, 1H), 7.26 (d, *J* = 8.2 Hz, 2H), 4.42 – 4.34 (m, 4H), 1.42 – 1.36 (m, 6H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 166.2, 165.9, 163.3, 140.5, 138.6, 137.8, 136.8, 132.8, 131.8, 130.4, 129.68, 129.66, 129.5, 129.4, 127.5, 127.0, 125.2, 125.1, 117.0, 61.2, 61.1, 14.3, 14.2.

3,4-Bis(4-fluorophenyl)isoquinolin-1(2*H*)-one (3af).[6]



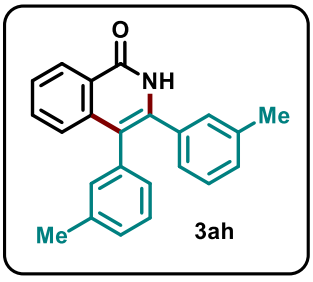
Yellow syrup (54 mg, 54%); eluent (25-30% ethyl acetate in hexane); **^{1}H NMR (500 MHz, DMSO-D_6)** δ 11.62 (brs, 1H), 8.31 (d, *J* = 7.7 Hz, 1H), 7.68 – 7.64 (m, 1H), 7.53 (t, *J* = 7.5 Hz, 1H), 7.30 – 7.26 (m, 2H), 7.22 – 7.12 (m, 5H), 7.09 (t, *J* = 8.9 Hz, 2H); **^{13}C NMR (125 MHz, DMSO-D_6)** δ 162.7 (d, $J_{C\text{-}F}$ = 244.1Hz), 161.7, 161.2 (d, $J_{C\text{-}F}$ = 242.5 Hz), 138.0, 133.7 (d, $J_{C\text{-}F}$ = 8.1 Hz), 132.6, 132.1 (d, $J_{C\text{-}F}$ = 8.4 Hz), 132.0 (d, $J_{C\text{-}F}$ = 2.5 Hz), 130.9, 126.6 (d, $J_{C\text{-}F}$ = 59.2 Hz), 125.1, 124.8, 115.2 (d, $J_{C\text{-}F}$ = 21.0 Hz), 114.7 (d, $J_{C\text{-}F}$ = 21.7 Hz), 114.6 (two carbons in the aromatic region are missing due to the overlap); **^{19}F NMR (470 MHz, $CDCl_3$)** δ –111.5, –114.2.

3,4-Bis(3,4-dimethoxyphenyl)isoquinolin-1(2*H*)-one (3ag).



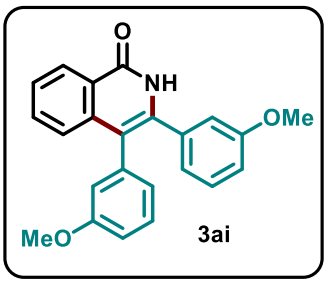
White solid (72.8 mg, 58%); eluent (35-40% ethyl acetate in hexane); m.p. 220 – 225 °C; **^{1}H NMR (500 MHz, $CDCl_3$)** δ 10.17 (brs, 1H), 8.42 (d, *J* = 7.4 Hz, 1H), 7.61 – 7.55 (m, 1H), 7.49 – 7.45 (m, 1H), 7.42 (d, *J* = 8.1 Hz, 1H), 6.88 (dd, *J* = 8.3, 2.0 Hz, 1H), 6.85 (d, *J* = 8.2 1H), 6.80 (d, *J* = 1.9 Hz, 1H), 6.76 (dd, *J* = 8.3, 2.3 Hz, 2H), 6.71 (d, *J* = 1.9 Hz, 1H), 3.89 (s, 3H), 3.86 (s, 3H), 3.72 (s, 3H), 3.67 (s, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 163.1, 149.0, 148.8, 148.2, 148.1, 139.0, 137.1, 132.6, 128.6, 127.4, 127.3, 126.3, 125.5, 124.8, 124.1, 121.8, 116.4, 114.8, 112.5, 111.0, 110.5, 55.9, 55.80, 55.76, 55.7.; **HRMS (ESI)** m/z calcd. for $C_{25}H_{23}NNaO_5$ $[M+Na]^+$: 440.1474, found: 440.1479.

3,4-Di-*m*-tolylisoquinolin-1(2*H*)-one (3ah).[10]



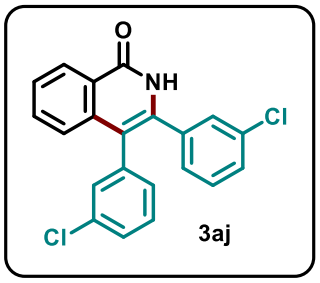
White solid (56.6 mg, 58%); eluent (25-30% ethyl acetate in hexane); **^{1}H NMR (500 MHz, $CDCl_3$)** δ 9.38 (brs, 1H), 8.47 (dd, *J* = 7.2, 1.0 Hz, 1H), 7.60 – 7.56 (m, 1H), 7.51 – 7.47 (m, 1H), 7.36 (d, *J* = 8.1 Hz, 1H), 7.20 (t, *J* = 7.6 Hz, 1H), 7.13 – 7.05 (m, 4H), 7.02 (s, 1H), 7.00 – 6.96 (m, 2H), 2.30 (s, 3H), 2.26 (s, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 162.7, 138.8, 138.0, 137.8, 137.0, 135.6, 134.9, 132.6, 132.4, 129.7, 129.3, 128.8, 128.2, 128.1, 128.0, 127.4, 126.44, 126.43, 125.7, 125.0, 117.2, 21.4, 21.3.

3,4-Bis(3-methoxyphenyl)isoquinolin-1(2*H*)-one (3ai).[11]



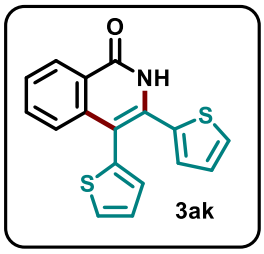
Yellow solid (73 mg, 68%); eluent (30-35% ethyl acetate in hexane); **^{1}H NMR (500 MHz, $CDCl_3$)** δ 10.02 (brs, 1H), 8.46 (d, *J* = 7.8 Hz, 1H), 7.62 – 7.57 (m, 1H), 7.53 – 7.47 (m, 1H), 7.41 (d, *J* = 8.1 Hz, 1H), 7.25 (t, *J* = 8 Hz, 1H), 7.18 (t, *J* = 7.9 Hz, 1H), 6.89 (d, *J* = 7.5 Hz, 1H), 6.87 – 6.84 (m, 1H), 6.83 – 6.78 (m, 3H), 6.75 – 6.73 (m, 1H), 3.71 (s, 3H), 3.63 (s, 3H). **^{13}C NMR (125 MHz, $CDCl_3$)** δ 163.2, 159.5, 159.0, 138.5, 137.3, 137.1, 135.9, 132.5, 129.3, 129.2, 127.3, 126.4, 125.6, 124.9, 124.2, 121.6, 117.2, 116.9, 115.2, 114.3, 112.9, 55.1, 55.1.

3,4-Bis(3-chlorophenyl)isoquinolin-1(2*H*)-one (3aj).[7]



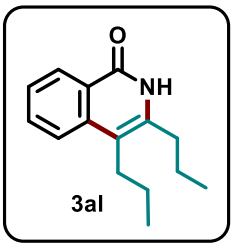
Yellow solid (74.7 mg, 68%); eluent (25-30% ethyl acetate in hexane); **^{1}H NMR (500 MHz, DMSO-D$_6$)** δ 11.70 (brs, 1H), 8.32 (d, *J* = 8.3 Hz, 1H), 7.70 – 7.65 (m, 1H), 7.55 (t, *J* = 7.5 Hz, 1H), 7.39 – 7.32 (m, 4H), 7.30 – 7.25 (m, 2H), 7.22 – 7.16 (m, 2H), 7.13 (d, *J* = 8.1 Hz, 1H). **^{13}C NMR (125 MHz, DMSO-D$_6$)** δ 161.5, 137.7, 137.46, 137.45, 136.1, 132.8, 132.7, 132.3, 131.3, 130.5, 130.0, 129.7, 129.5, 128.6, 128.3, 127.3, 126.8, 126.6, 125.1, 124.7, 114.5.

3,4-Di(thiophen-2-yl)isoquinolin-1(2*H*)-one (3ak).[8]



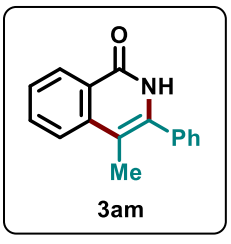
White solid (74.4 mg, 80%); eluent (35-40% ethyl acetate in hexane); **^{1}H NMR (500 MHz, DMSO-D$_6$)** δ 11.57 (brs, 1H), 8.28 (dd, *J* = 7.6, 1.0 Hz, 1H)), 7.76 – 7.72 (m, 1H), 7.70 – 7.65 (m, 1H), 7.64 – 7.61 (m, 1H), 7.60 – 7.58 (m, 1H), 7.52 (t, *J* = 7.6 Hz, 1H), 7.23 – 7.18 (m, 2H), 7.12 (d, *J* = 3.4 Hz, 1H) , 7.06 – 7.02 (m, 1H); **^{13}C NMR (125 MHz, DMSO-D$_6$)** δ 161.6, 138.6, 135.8, 134.7, 134.1, 132.8, 131.2, 129.9, 129.3, 128.6, 127.7, 126.6, 126.4, 125.0, 124.6, 107.1; (one carbon in the aromatic region is missing due to the overlap).

3,4-Dipropylisoquinolin-1(2*H*)-one (3al).[1]



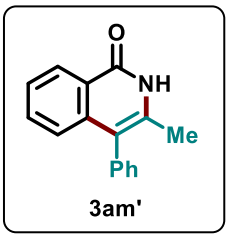
Yellow solid (41.3 mg, 60%); eluent (30-35% ethyl acetate in hexane); **^{1}H NMR (500 MHz, CDCl$_3$)** δ 11.73 (brs, 1H), 8.48 (d, *J* = 8.2 Hz, 1H), 7.70 – 7.65 (m, 2H), 7.48 – 7.41 (m, 1H), 2.74 – 2.67 (m, 4H), 1.81 – 1.72 (m, 2H), 1.65 – 1.56 (m, 2H), 1.11 – 1.01 (m, 6H); **^{13}C NMR (125 MHz, CDCl$_3$)** δ 163.9, 138.5, 138.4, 132.2, 127.6, 125.2, 125.0, 123.0, 112.9, 32.8, 28.5, 23.6, 22.8, 14.3, 14.0. **3-Ethyl-4-phenylisoquinolin-1(2*H*)-one (3na)**

4-Methyl-3-phenylisoquinolin-1(2H)-one (3ma)[12]



White solid (25.4 mg, 36%); eluent (20-25% ethyl acetate in hexane); **^{1}H NMR (500 MHz, CDCl$_3$)** δ 9.30 (s, 1H), 8.40 (d, *J* = 8.0 Hz, 1H), 7.74 – 7.67 (m, 2H), 7.55 – 7.35 (m, 6H), 2.23 (s, 3H); **^{13}C NMR (125 MHz, CDCl$_3$)** δ 162.4, 138.9, 136.5, 135.0, 133.2, 129.3, 129.22, 128.9, 127.6, 126.7, 124.5, 123.8, 110.4, 13.9.

3-Methyl-4-phenylisoquinolin-1(2*H*)-one (3ma')[12]

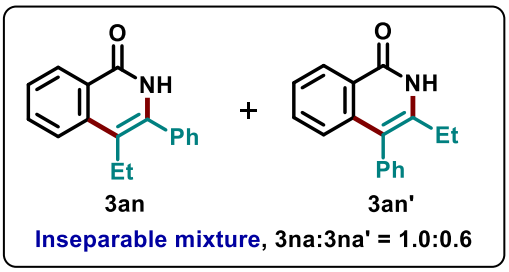


3am'

White solid (16.9 mg, 24%); eluent (30-35% ethyl acetate in hexane); **^{1}H NMR (500 MHz, $CDCl_3$)** δ 10.91 (s, 1H), 8.39 (d, *J* = 8.1 Hz, 1H), 7.51 – 7.34 (m, 5H), 7.24 – 7.17 (m, 2H), 7.09 (d, *J* = 8.2 Hz, 1H), 2.16 (s, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 163.9, 139.2, 136.4, 134.7, 132.5, 131.1, 128.8, 127.6, 127.3, 125.7, 124.9, 124.3, 117.3, 17.9.

3-Ethyl-4-phenylisoquinolin-1(2*H*)-one (3na)[13]

4-Ethyl-3-phenylisoquinolin-1(2*H*)-one (3na')[13]

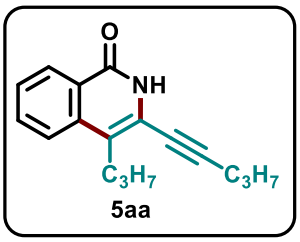


3an 3an'

Inseparable mixture, 3na:3na' = 1.0:0.6

Compounds **3na** and **3na'** were obtained as an inseparable mixture in ratio 1.0:0.6. Yellow solid (50.0 mg, 67%); eluent (30-35% ethyl acetate in hexane); **^{1}H NMR (500 MHz, $CDCl_3$)** δ 11.79 (brs, 0.6H), 10.19 (brs, 1H), 8.46 (dd, *J* = 8.1, 0.9 Hz, 1H), 8.36 (dd, *J* = 8.0, 0.8 Hz, 0.6H), 7.78 (d, *J* = 8.1 Hz, 1H), 7.76 – 7.70 (m, 1H), 7.54 – 7.35 (m, 9H), 7.29 – 7.24 (m, 1.2H), 7.07 (d, *J* = 8.1 Hz, 0.6H), 2.68 (q, *J* = 7.5 Hz, 2H), 2.51 (q, *J* = 7.6 Hz, 1.2H), 1.25 (t, *J* = 7.6 Hz, 1.8H), 1.20 (t, *J* = 7.5 Hz, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 164.2, 162.7, 140.5, 139.1, 137.7, 137.0, 136.3, 135.2, 132.6, 132.2, 131.0, 129.0, 128.6, 127.9, 127.4, 127.1, 126.1, 125.8, 125.4, 125.0, 124.3, 123.6, 116.2, 115.6, 24.8, 20.4, 15.3, 13.9.

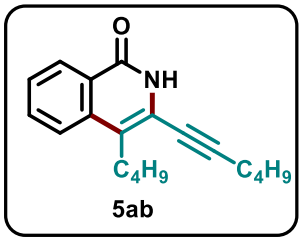
3-(Pent-1-yn-1-yl)-4-propylisoquinolin-1(2*H*)-one (5aa).[14]



5aa

Yellow solid (43.4 mg, 57%); eluent (20-25% ethyl acetate in hexane); **^{1}H NMR (500 MHz, $CDCl_3$)** δ 10.10 (brs, 1H), 8.44 (d, *J* = 7.8 Hz, 1H), 7.72 – 7.65 (m, 2H), 7.50 – 7.44 (m, 1H), 2.90 – 2.84 (m, 2H), 2.49 (t, *J* = 7.0 Hz, 2H), 1.74 – 1.60 (m, 4H), 1.10 (t, *J* = 7.3 Hz, 3H), 1.01 (t, *J* = 7.4 Hz, 3H). **^{13}C NMR (125 MHz, $CDCl_3$)** δ 162.7, 137.2, 132.4, 127.9, 126.5, 126.2, 123.5, 120.8, 120.7, 97.7, 74.4, 30.4, 23.0, 21.8, 21.5, 14.1, 13.5.

4-Butyl-3-(hex-1-yn-1-yl)isoquinolin-1(2*H*)-one (5ab).[14]

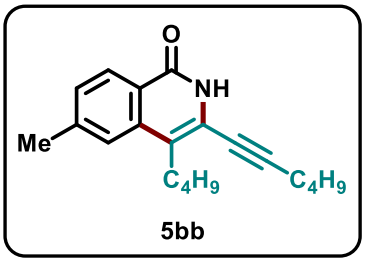


5ab

Yellow solid (51.5 mg, 61%); eluent (20-25% ethyl acetate in hexane); **^{1}H NMR (500 MHz, $CDCl_3$)** δ 9.06 (brs, 1H), 8.43 (d, *J* = 8.0 Hz, 1H), 7.72 – 7.66 (m, 2H), 7.50 – 7.46 (m, 1H), 2.91 – 2.85 (m, 2H), 2.50 (t, *J* = 6.9 Hz, 2H), 1.65 – 1.56 (m, 4H), 1.54 – 1.49 (m, 2H), 1.48 – 1.41 (m, 2H), 0.99 – 0.95 (m, 6H); **^{13}C NMR (125**

MHz, $CDCl_3$) δ 162.9, 137.2, 132.4, 127.9, 126.4, 126.3, 123.5, 120.9, 120.8, 97.8, 74.2, 31.9, 30.4, 28.2, 22.7, 22.0, 19.2, 13.9, 13.6.

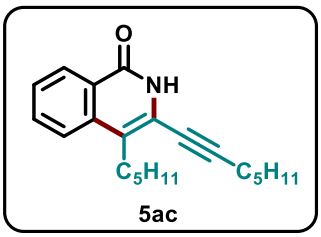
4-butyl-3-(hex-1-yn-1-yl)-7-methylisoquinolin-1(2*H*)-one (5bb).[14]



5bb

Yellow solid (48.8 mg, 55%); eluent (20-25% ethyl acetate in hexane); **^{1}H NMR (500 MHz, $CDCl_3$)** δ 10.31 (brs, 1H), 8.33 (d, *J* = 8.2 Hz, 1H), 7.43 (s, 1H), 7.28 (dd, *J* = 8.2, 0.8 Hz, 1H), 2.90 – 2.83 (m, 2H), 2.54 – 2.48 (m, 5H), 1.68 – 1.61 (m, 2H), 1.61 – 1.50 (m, 4H), 1.49 – 1.40 (m, 2H), 0.99 – 0.94 (m, 6H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 162.6, 142.9, 137.3, 128.0, 127.9, 124.0, 123.3, 120.7, 120.7, 97.6, 74.3, 31.9, 30.4, 28.2, 22.7, 22.2, 22.0, 19.2, 14.0, 13.6.

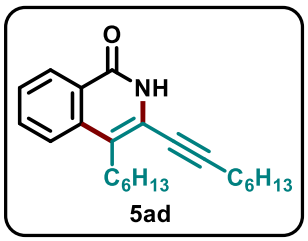
3-(Hept-1-yn-1-yl)-4-pentylisoquinolin-1(2*H*)-one (5ac).[14]



5ac

Yellow solid (35.5 mg, 38%); eluent (20-25% ethyl acetate in hexane); **^{1}H NMR (500 MHz, $CDCl_3$)** δ 10.24 (brs, 1H), 8.45 (d, *J* = 8.1 Hz, 1H), 7.71 – 7.65 (m, 2H), 7.49 – 7.44 (m, 1H), 2.91 – 2.85 (m, 2H), 2.51 (t, *J* = 7.1 Hz, 2H), 1.71 – 1.65 (m, 2H), 1.65 – 1.57 (m, 2H), 1.51 – 1.45 (m, 2H), 1.44 – 1.33 (m, 6H), 0.95 – 0.88 (m, 6H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 162.6, 137.2, 132.5, 127.9, 126.5, 126.3, 123.5, 121.0, 120.6, 97.9, 74.2, 31.8, 31.1, 29.5, 28.5, 28.1, 22.5, 22.2, 19.5, 14.0, 13.9.

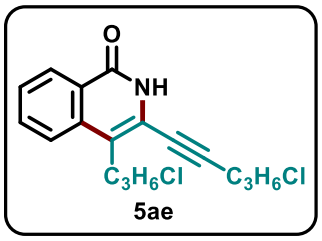
4-Hexyl-3-(oct-1-yn-1-yl)isoquinolin-1(2*H*)-one (5ad).[14]



5ad

Yellow solid (40.5 mg, 40%); eluent (20-25% ethyl acetate in hexane); **^{1}H NMR (500 MHz, $CDCl_3$)** δ 9.37 (brs, 1H), 8.43 (d, *J* = 8.1 Hz, 1H), 7.72 – 7.65 (m, 2H), 7.47 (ddd, *J* = 8.1, 6.5, 1.8 Hz, 1H), 2.90 – 2.85 (m, 2H), 2.49 (t, *J* = 7.1 Hz, 2H), 1.69 – 1.56 (m, 4H), 1.45 (m, 4H), 1.37 – 1.29 (m, 8H), 0.94 – 0.83 (m, 6H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 162.4, 137.2, 132.5, 128.0, 126.5, 126.3, 123.5, 121.1, 120.5, 97.9, 74.2, 31.7, 31.3, 29.8, 29.3, 28.6, 28.5, 28.3, 22.7, 22.5, 19.5, 14.09, 14.07.

3-(5-Chloropent-1-yn-1-yl)-4-(3-chloropropyl)isoquinolin-1(2*H*)-one (5ae).[14]

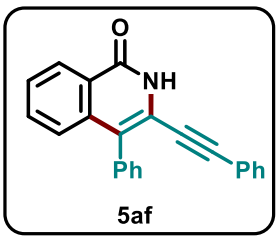


5ae

Yellow solid (56.1 mg, 58%); eluent (25-30% ethyl acetate in hexane); **^{1}H NMR (500 MHz, $CDCl_3$)** δ 10.87 (brs, 1H), 8.47 (d, *J* = 8.1 Hz, 1H), 7.77 – 7.68 (m, 2H), 7.56 – 7.48 (m, 1H), 3.81 (t, *J* = 6.4 Hz, 2H), 3.65 (t, *J* = 6.2 Hz, 2H), 3.11 – 3.06 (m, 2H), 2.76 (t, *J*

= 6.8 Hz, 2H), 2.19 – 2.13 (m, 2H), 2.13 – 2.04 (m, 2H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 162.9, 136.8, 132.8, 128.1, 127.0, 126.3, 123.3, 121.0, 119.6, 96.3, 74.7, 44.8, 43.6, 32.5, 30.9, 25.8, 17.0.

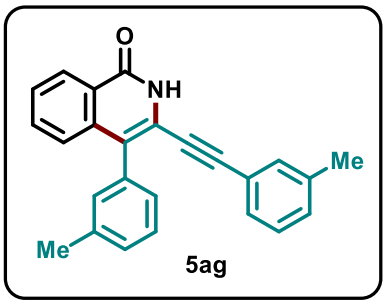
4-Phenyl-3-(phenylethynyl)isoquinolin-1(2*H*)-one (3af).[1]



5af

Yellow solid (65.6 mg, 68%); eluent (25-30% ethyl acetate in hexane); **^{1}H NMR (500 MHz, DMSO-D_6),** δ 11.94 (brs, 1H), 8.30 (d, *J* = 7.9 Hz, 1H), 7.68 (t, *J* = 7.6 Hz, 1H), 7.59 – 7.52 (m, 3H), 7.52 – 7.44 (m, 3H), 7.41 – 7.34 (m, 3H), 7.26 (d, *J* = 8.1 Hz, 1H), 7.22 – 7.18 (m, 2H); **^{13}C NMR (125 MHz, DMSO-D_6)** δ 161.2, 136.9, 135.3, 132.9, 131.2, 131.0, 129.6, 128.9, 128.5, 128.1, 127.5, 127.2, 126.2, 125.3, 122.7, 121.2, 121.1, 95.1, 83.8.

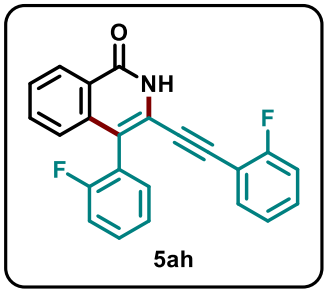
4-(*m*-Tolyl)-3-(o-tolylethynyl)isoquinolin-1(2*H*)-one (5ag).



5ag

Yellow solid (69.2 mg, 66%); eluent (30-35% ethyl acetate in hexane); m.p. 178 – 180 °C; **^{1}H NMR (500 MHz, DMSO-D_6)** δ 11.90 (brs, 1H), 8.29 (dd, *J* = 8.1, 1.1 Hz, 1H), 7.72 – 7.66 (m, 1H), 7.56 (t, *J* = 7.5 Hz, 1H), 7.43 (t, *J* = 7.6 Hz, 1H), 7.33 – 7.20 (m, 6H), 7.03 – 7.00 (m, 2H), 2.40 (s, 3H), 2.26 (s, 3H); **^{13}C NMR (125 MHz, DMSO-D_6)** δ 161.1, 138.1, 137.5, 136.8, 135.1, 132.7, 131.5, 131.4, 130.2, 128.7, 128.5, 128.24, 128.17, 128.0, 127.3, 127.1, 126.1, 125.3, 122.6, 121.1, 121.0, 95.3, 83.6, 21.0, 20.7; **HRMS (ESI)** m/z calcd. for $C_{25}H_{20}NO$ $[M+H]^+$: 350.1545, found: 350.1549.

4-(2-Fluorophenyl)-3-[(2-fluorophenyl)ethynyl]isoquinolin-1(2*H*)-one (5ah).

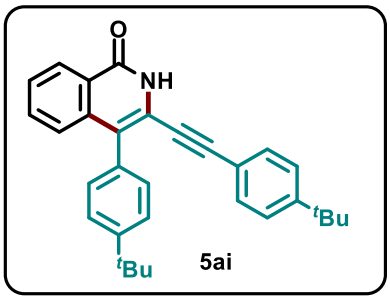


5ah

Yellow solid (77.2 mg, 72%); eluent (30-35% ethyl acetate in hexane); m.p. 227 – 229 °C; **^{1}H NMR (500 MHz, DMSO-D_6)** δ 12.04 (brs, 1H), 8.31 (dd, *J* = 8.5, 1.0 Hz, 1H), 7.73 – 7.69 (m, 1H), 7.62 – 7.54 (m, 2H), 7.52 – 7.44 (m, 2H), 7.41 – 7.35 (m, 2H), 7.34 – 7.30 (td, *J* = 7.5, 1.6 Hz, 1H), 7.25 (t, *J* = 9.0 Hz, 1H), 7.22 (t, *J* = 7.6 Hz, 1H), 7.14 (d, *J* = 8.1 Hz, 1H); **^{13}C NMR (125 MHz, DMSO-D_6)** δ 162.5 (d, $J_{C\text{-}F}$ = 252.1 Hz), 161.1, 159.8 (d, $J_{C\text{-}F}$ = 245.0 Hz), 136.3, 133.3, 133.2, 132.9, 131.9 (d, $J_{C\text{-}F}$ = 7.6 Hz), 130.7 (d, $J_{C\text{-}F}$ = 8.0 Hz), 127.6, 127.1, 126.1, 124.8, 124.7, 124.6,

122.3 (d, $J_{C\text{-}F}$ = 16.3 Hz), 121.8, 116.7, 115.8 (d, $J_{C\text{-}F}$ = 11.1 Hz), 115.6 (d, $J_{C\text{-}F}$ = 13.0 Hz), 109.3 (d, $J_{C\text{-}F}$ = 15.4 Hz), 88.7, 87.4 (d, $J_{C\text{-}F}$ = 1.8 Hz); **^{19}F NMR (470 MHz, $CDCl_3$)** δ –109.3, –113.6; **HRMS (ESI)** m/z calcd. for $C_{23}H_{14}F_2NO$ $[M+H]^+$: 358.1043, found: 358.1044.

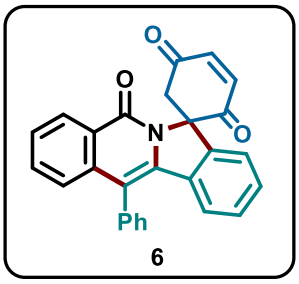
4-[4-(*tert*-Butyl)phenyl]-3-[(4-(*tert*-butyl)phenyl)ethynyl]isoquinolin-1(2*H*)-one (5ai).



5ai

Yellow solid (70.4 mg, 54%); eluent (30-35% ethyl acetate in hexane); m.p. 268 – 270 °C; **^{1}H NMR (500 MHz, $CDCl_3$)** δ 9.51 (brs, 1H), 8.48 (d, *J* = 7.8 Hz, 1H), 7.62 – 7.58 (m, 1H), 7.55 – 7.49 (m, 3H), 7.45 (d, *J* = 8.1 Hz, 1H), 7.40 (d, *J* = 8.2 Hz, 2H), 7.29 (d, *J* = 8.3 Hz, 2H), 7.16 – 7.12 (m, 2H), 1.43 (s, 9H), 1.29 (s, 9H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 162.8, 152.4, 150.7, 137.7, 132.5, 131.3, 130.8, 127.6, 127.0, 126.0, 125.8, 125.2, 125.1, 124.0, 121.3, 118.8, 96.3, 83.0, 34.8, 34.7, 31.4, 31.1; (one carbon is missing in the aromatic region due to the overlap). **HRMS (ESI)** m/z calcd. for $C_{31}H_{32}NO$ $[M+H]^+$: 434.2484, found: 434.2484.

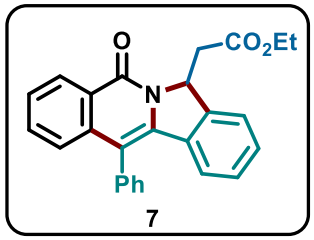
12'-Phenyl-5'*H*-spiro[cyclohexane-1,7'-isoindolo[2,1-*b*]isoquinolin]-3-ene-2,5,5'-trione (6).[2]



6

Yellow solid (60.6 mg, 75%); eluent (30-35% ethyl acetate in hexane); **^{1}H NMR (500 MHz, $CDCl_3$)** δ 8.46 (dd, *J* = 8.2, 1.0 Hz, 1H), 7.65 – 7.59 (m, 4H), 7.52 (t, *J* = 7.5 Hz, 1H), 7.47 – 7.41 (m, 2H), 7.29 – 7.25 (m, 1H), 7.23 (d, *J* = 8.2 Hz, 1H), 7.20 (d, *J* = 7.7 Hz, 1H), 7.17 (s, 2H), 7.15 – 7.11 (m, 1H), 6.41 (d, *J* = 8.2 Hz, 1H), 4.91 (d, *J* = 16.0 Hz, 1H), 2.97 (d, *J* = 16.0 Hz, 1H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 195.2, 189.2, 160.3, 142.8, 141.4, 140.0, 138.9, 136.9, 134.5, 133.2, 132.7, 130.9, 130.8, 129.8, 129.7, 129.6, 129.5, 128.7, 127.5, 126.9, 125.6, 125.1, 125.0, 121.0, 115.3, 74.5, 45.9.

Ethyl 2-(5-oxo-12-phenyl-5,7-dihydroisoindolo[2,1-b]isoquinolin-7-yl)acetate (7).[3]

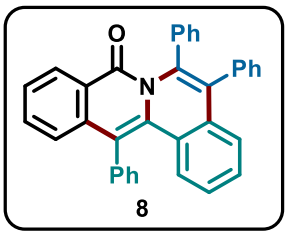


7

Yellow solid (24.0 mg, 60%); eluent (30-35% ethyl acetate in hexane); **^{1}H NMR (500 MHz, $CDCl_3$)** δ 8.54 (dd, *J* = 7.7, 1.0 Hz, 1H), 7.63 – 7.43 (m, 7H), 7.37 – 7.30 (m, 2H), 7.20 (d, *J* = 8.0 Hz, 1H), 7.09 (t, *J* = 7.7 Hz, 1H), 6.38 (d, *J* = 7.9 Hz, 1H), 6.01 (dd, *J* = 7.7, 3.6 Hz, 1H), 4.11 (q, *J* = 7.1 Hz, 2H), 3.73 (dd, *J* = 16.1, 3.7 Hz, 1H), 3.06 (dd, *J* = 16.0, 7.7 Hz, 1H), 1.15 (t, *J* = 7.1 Hz, 3H); **^{13}C NMR (125 MHz, $CDCl_3$)**

δ 170.4, 160.9, 141.6, 138.7, 137.9, 135.1, 133.5, 132.1, 131.1, 130.9, 129.5, 129.4, 128.43, 128.38, 127.3, 126.3, 125.2, 124.8, 123.9, 122.8, 114.5, 60.6, 56.0, 36.6, 14.0 (one carbon is missing in the aromatic region due to the overlap).

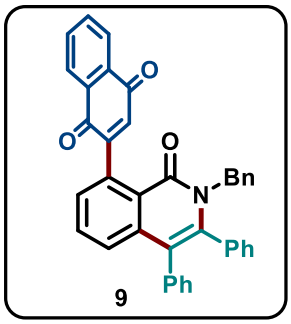
5,6,13-Triphenyl-8H-isoquinolino[3,2-a]isoquinolin-8-one (8).[4]



8

Yellow solid (33.2 mg, 70%); eluent (30-35% ethyl acetate in hexane); **^{1}H NMR (500 MHz, $CDCl_3$)** δ 8.25 (d, *J* = 8.0 Hz, 1H), 7.62 – 7.57 (m, 1H), 7.56 – 7.49 (m, 5H), 7.43 (t, *J* = 7.5 Hz, 1H), 7.34 (d, *J* = 8.3 Hz, 1H), 7.28 – 7.20 (m, 3H), 7.17 – 7.10 (m, 5H), 7.09 – 7.04 (m, 5H), 6.89 – 6.84 (m, 1H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 162.2, 138.5, 137.1, 136.2, 136.1, 133.7, 133.0, 132.3, 132.1, 131.4, 129.7, 129.0, 128.8, 128.4, 128.1, 127.9, 127.6, 127.4, 127.1, 126.9, 126.8, 126.7, 126.4, 126.3, 125.7, 125.6, 125.5, 116.9 (one carbon is missing in the aromatic region due to the overlap).

2-(2-Benzyl-1-oxo-3,4-diphenyl-1,2-dihydroisoquinolin-8-yl)naphthalene-1,4-dione (9).



9

Brown solid (11 mg, 20%); eluent (30-35% ethyl acetate in hexane); **^{1}H NMR (500 MHz, $CDCl_3$)** δ 8.17 – 8.13 (m, 1H), 8.12 – 8.08 (m, 1H), 7.75 – 7.69 (m, 2H), 7.65 – 7.60 (m, 1H), 7.44 – 7.39 (m, 1H), 7.31 (dd, *J* = 8.2, 1.1 Hz, 1H), 7.21 – 7.12 (m, 6H), 7.12 – 7.00 (m, 5H), 6.92 (s, 1H), 6.88 – 6.74 (m, 4H), 5.08 (br s, 2H); **^{13}C NMR (125 MHz, $CDCl_3$)** δ 185.6, 183.9, 161.9, 155.0, 142.1, 138.4, 137.1, 136.2, 136.0, 133.9, 133.5, 133.4, 133.3, 132.6, 131.9, 131.5, 130.3, 130.2, 128.5, 128.20, 128.12, 128.1, 127.9, 127.6, 127.6, 127.5, 126.94 126.87, 126.8, 126.8, 126.1, 123.9, 119.6, 49.1; **HRMS (ESI)** m/z calcd. for $C_{38}H_{25}NaNO_3$ $[M+Na]^+$: 566.1732, found: 566.1737.

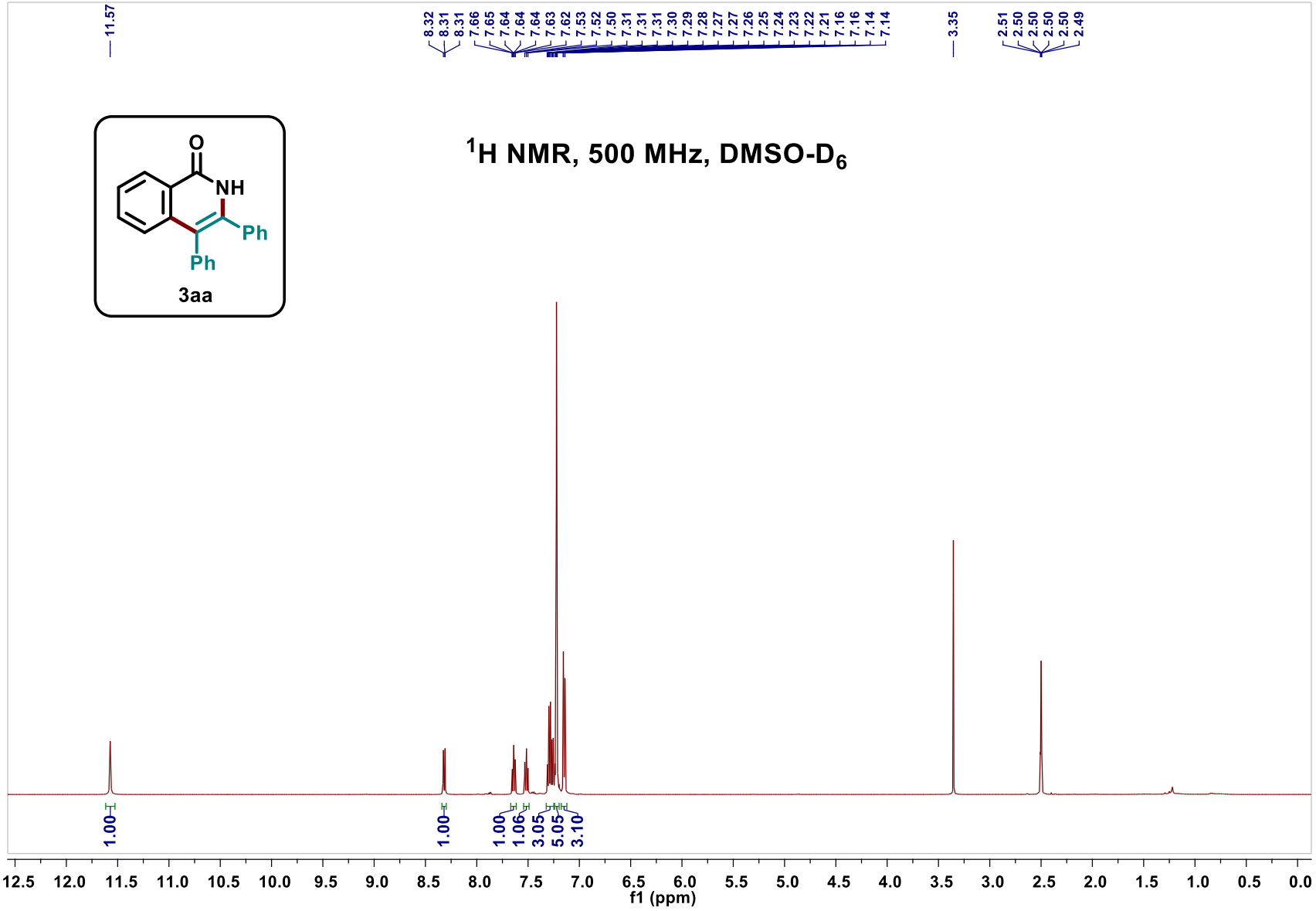
11. References

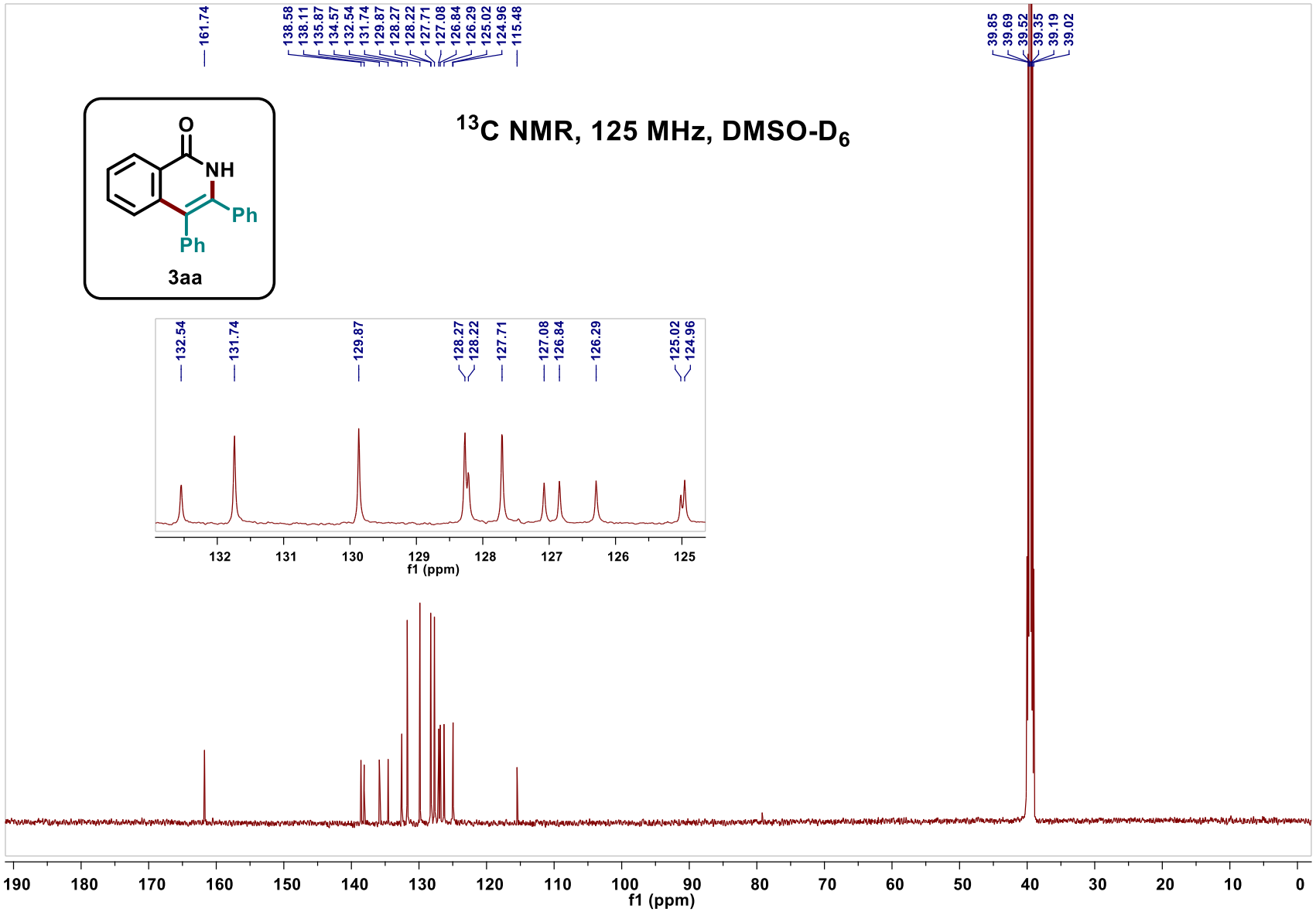
1. J. Liu, X. Jia and L. Huang, Org. Lett., 2022, **24**, 6772-6776.
2. T. Zhou, L. Li, B. Li, H. Song and B. Wang, *Org. Lett.*, 2015, **17**, 4204–4207.
3. F. Wang, G.-Y. Song, Z.-Y. Du and X.-W. Li, *J. Org. Chem.*, 2011, **76**, 2926–2932.
4. B. Li, H. Feng, N. Wang, J. Ma, H. Song, S. Xu and B. Wang, *Chem.–Eur. J.*, 2012, **18**, 12873–12879.

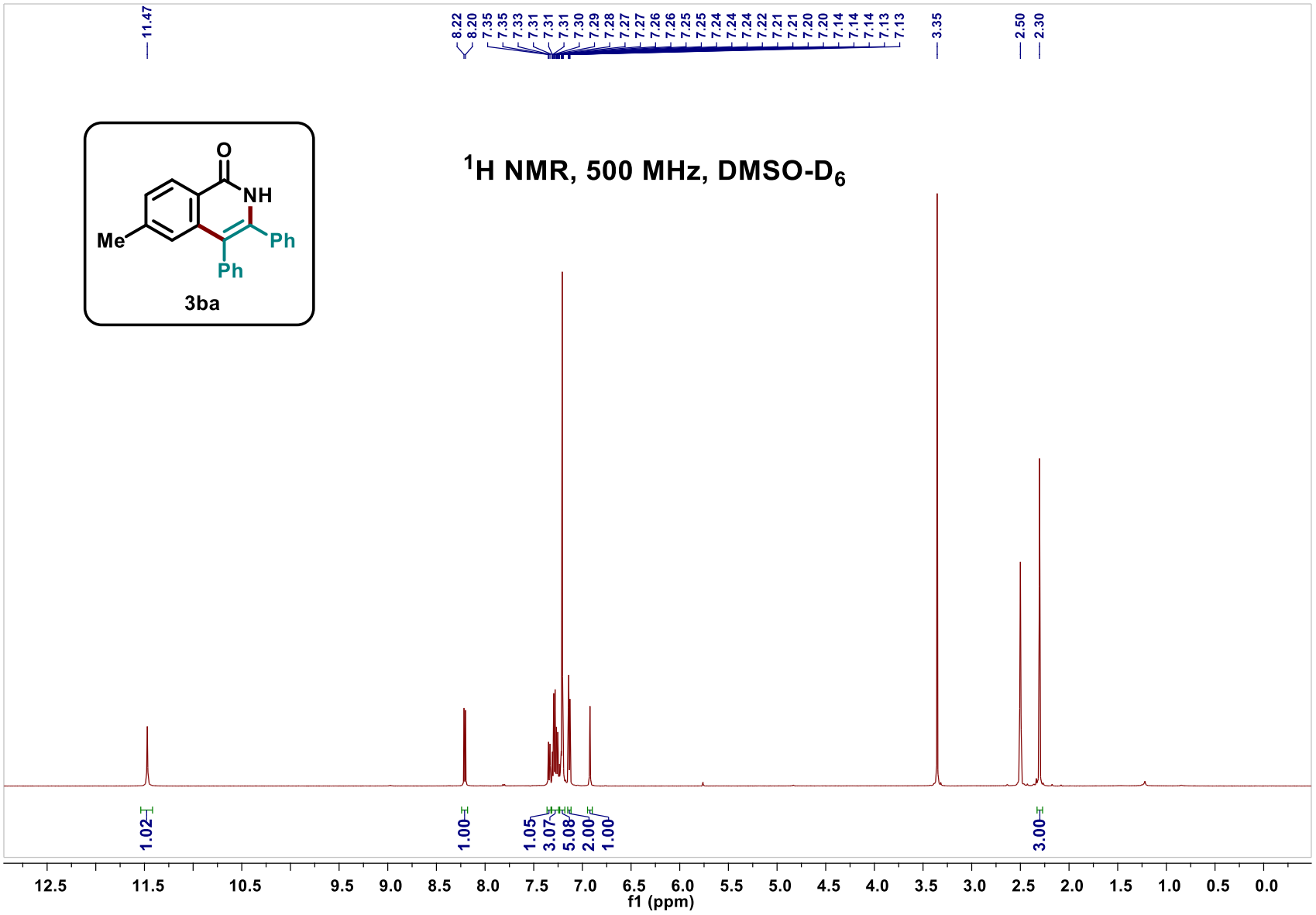
5. T. K. Hyster and T. Rovis, *J. Am. Chem. Soc.*, 2010, **132**, 10565–10569.
6. X. Yu, K. Chen, S. Guo, P. Shi, C. Song and J. Zhu, *Org. Lett.* 2017, ***19***, 5348–5351.
7. X. Li, R. Zhang, Y. Qi, Q. Zhao and T. Yao, *Org. Chem. Front.*, 2021, **8**, 1190–1196.
8. S. Wang, L. Yao, J.-S. Wang, J. Ying and X.-F. Wu, *Mol. Catal.*, 2022, **524**, 112303.
9. B. Su, J.-B. Wei, W.-L. Wu and Z.-J. Shi, *ChemCatChem*, 2015, **7**, 2986−2990.
10. M. Liu, J.-L. Niu, D. Yang and M.-P. Song, *J. Org. Chem*., 2020, **85**, 4067−4078.
11. E. Petrova, D. Rasina and A. Jirgensons, *Eur. J. Org. Chem*., 2017, 1773−1779.
12. M. Liu, J. L. Niu, D. Yang and M. P. Song, *J. Org. Chem.*, 2020, **85**, 4067–4078.
13. X. Yu, K. Chen, S. Guo, P. Shi, C. Song and J. Zhu, *Org. Lett.*, 2017, **19**, 5348–5351.
14. A. Ghosh, G.T. Sapkal and A. B. Pawar*, J. Org. Chem*., 2023, **88**, 4704–4719.

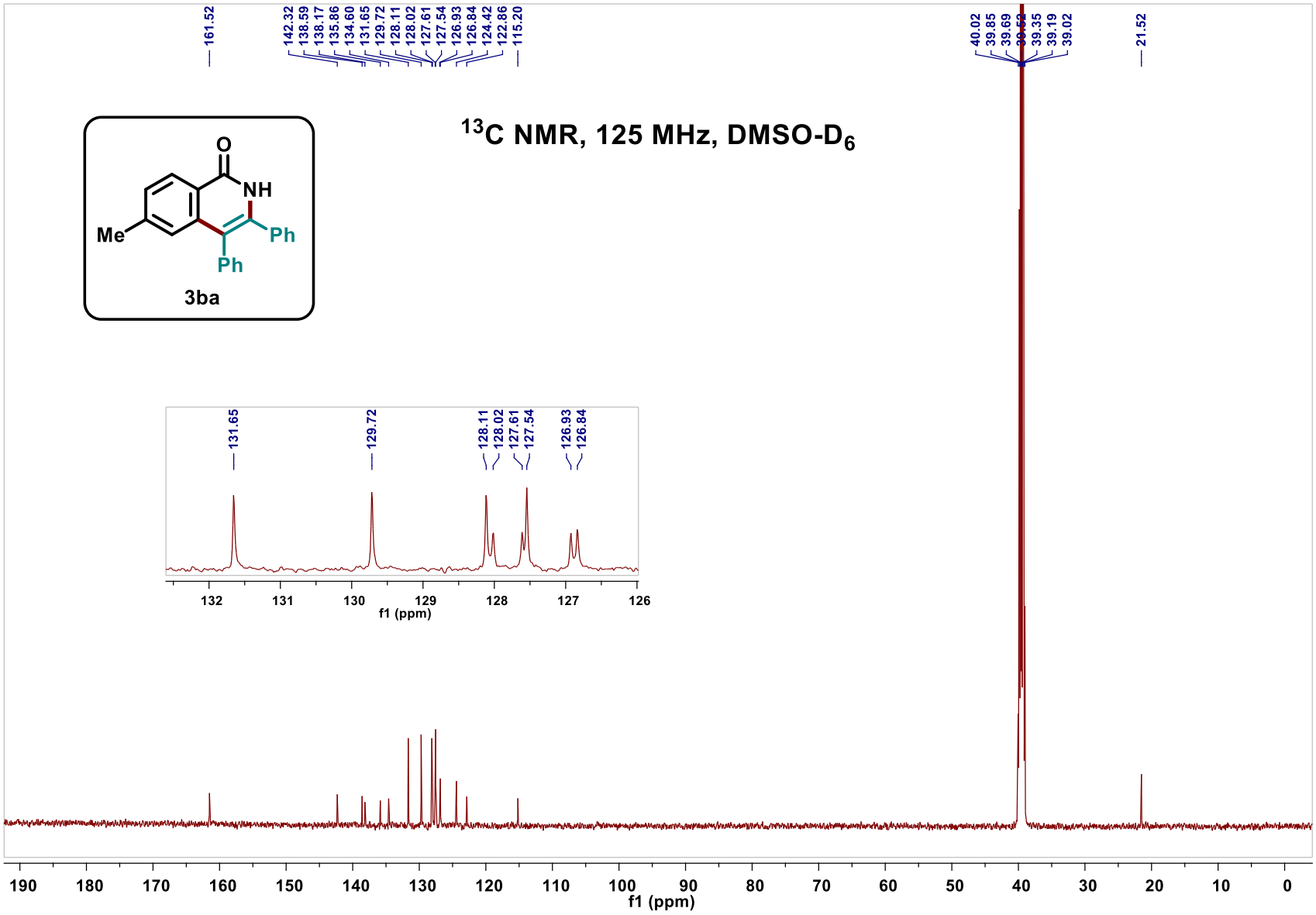
Appendix

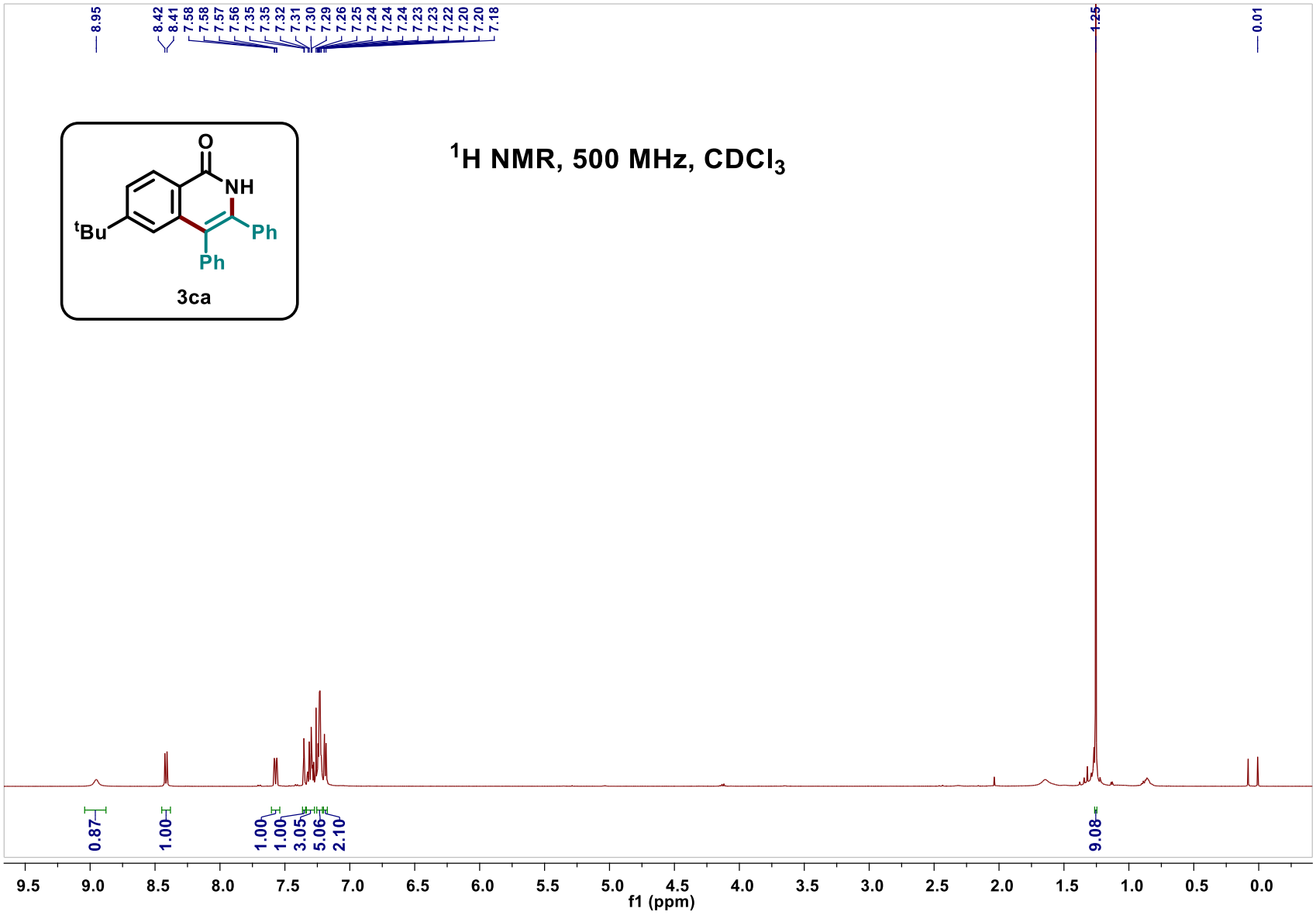
Spectral Copies of ^{1}H and ^{13}C NMR of Compounds Obtained in this Study

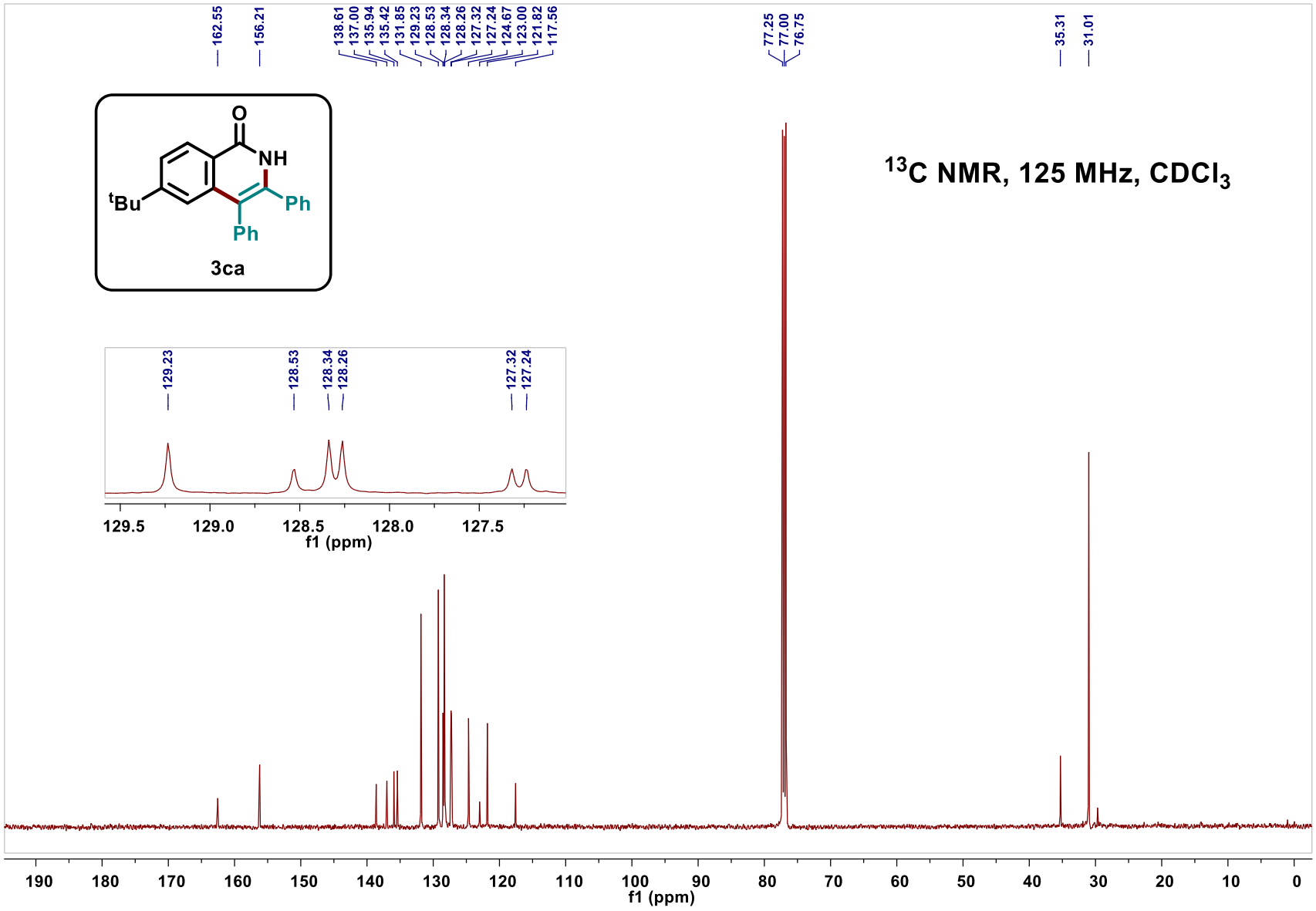

11.57
8.32
8.31
8.31
7.66
7.65
7.64
7.64
7.64
7.63
7.62
7.53
7.52
7.50
7.31
7.31
7.31
7.30
7.29
7.28
7.27
7.27
7.26
7.25
7.24
7.23
7.22
7.21
7.16
7.16
7.14
7.14
3.35
2.51
2.50
2.50
2.50
2.50
2.49
O
NH
Ph
Ph
3aa
1H NMR, 500 MHz, DMSO-D6
1.00
1.00
1.00
1.06
3.05
5.05
3.10
12.5
12.0
11.5
11.0
10.5
10.0
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
0.0
f1 (ppm)


161.74
138.58
138.11
135.87
134.57
132.54
131.74
129.87
128.27
128.22
127.71
127.08
126.84
126.29
125.02
124.96
115.48
39.85
39.69
39.52
39.35
39.19
39.02
13C NMR, 125 MHz, DMSO-D6
O
NH
Ph
Ph
3aa
f1 (ppm)
f1 (ppm)


11.47
8.22
8.20
7.35
7.33
7.31
7.31
7.31
7.30
7.29
7.28
7.27
7.27
7.26
7.25
7.25
7.24
7.24
7.24
7.22
7.21
7.21
7.20
7.20
7.14
7.14
7.14
7.13
7.13
3.35
2.50
2.30
O
NH
Me
Ph
Ph
3ba
1H NMR, 500 MHz, DMSO-D6
1.02
1.00
1.05
3.07
5.08
2.00
1.00
3.00
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


161.52
142.32
138.59
138.17
135.86
134.60
131.65
129.72
128.11
128.02
127.61
127.54
126.93
126.84
124.42
122.86
115.20
40.02
39.85
39.69
39.52
39.35
39.19
39.02
21.52
13C NMR, 125 MHz, DMSO-D6
O
NH
Me
Ph
Ph
3ba
131.65
129.72
128.11
128.02
127.61
127.54
126.93
126.84
132
131
130
129
128
127
126
f1 (ppm)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.95
8.42
8.41
7.58
7.57
7.56
7.35
7.35
7.32
7.31
7.30
7.29
7.26
7.25
7.24
7.24
7.24
7.23
7.23
7.22
7.20
7.20
7.18
1.25
0.01
O
NH
tBu
Ph
Ph
3ca
1H NMR, 500 MHz, CDCl3
0.87
1.00
1.00
1.00
3.05
5.06
2.10
9.08
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


162.55
156.21
138.61
137.00
135.94
135.42
131.85
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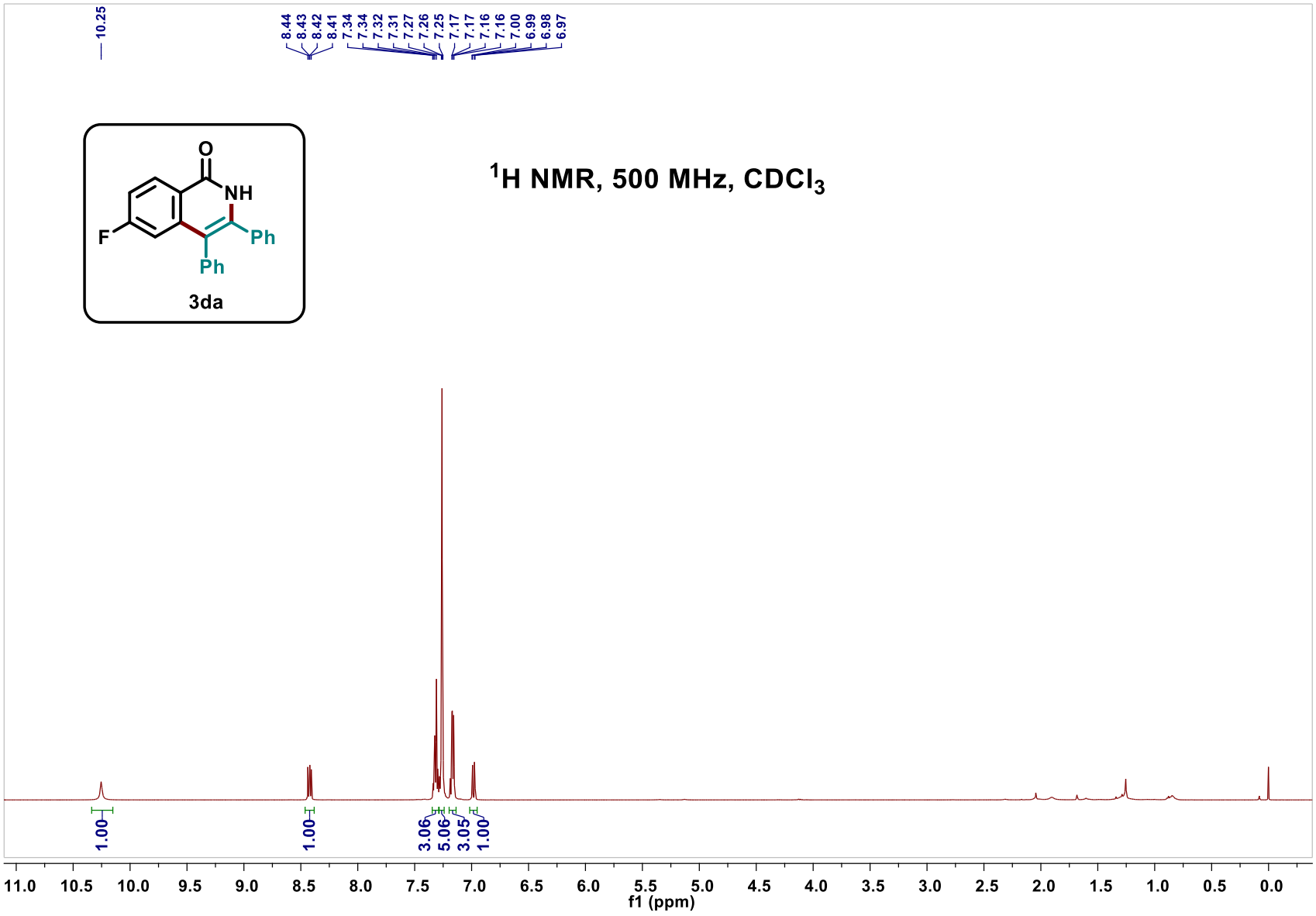
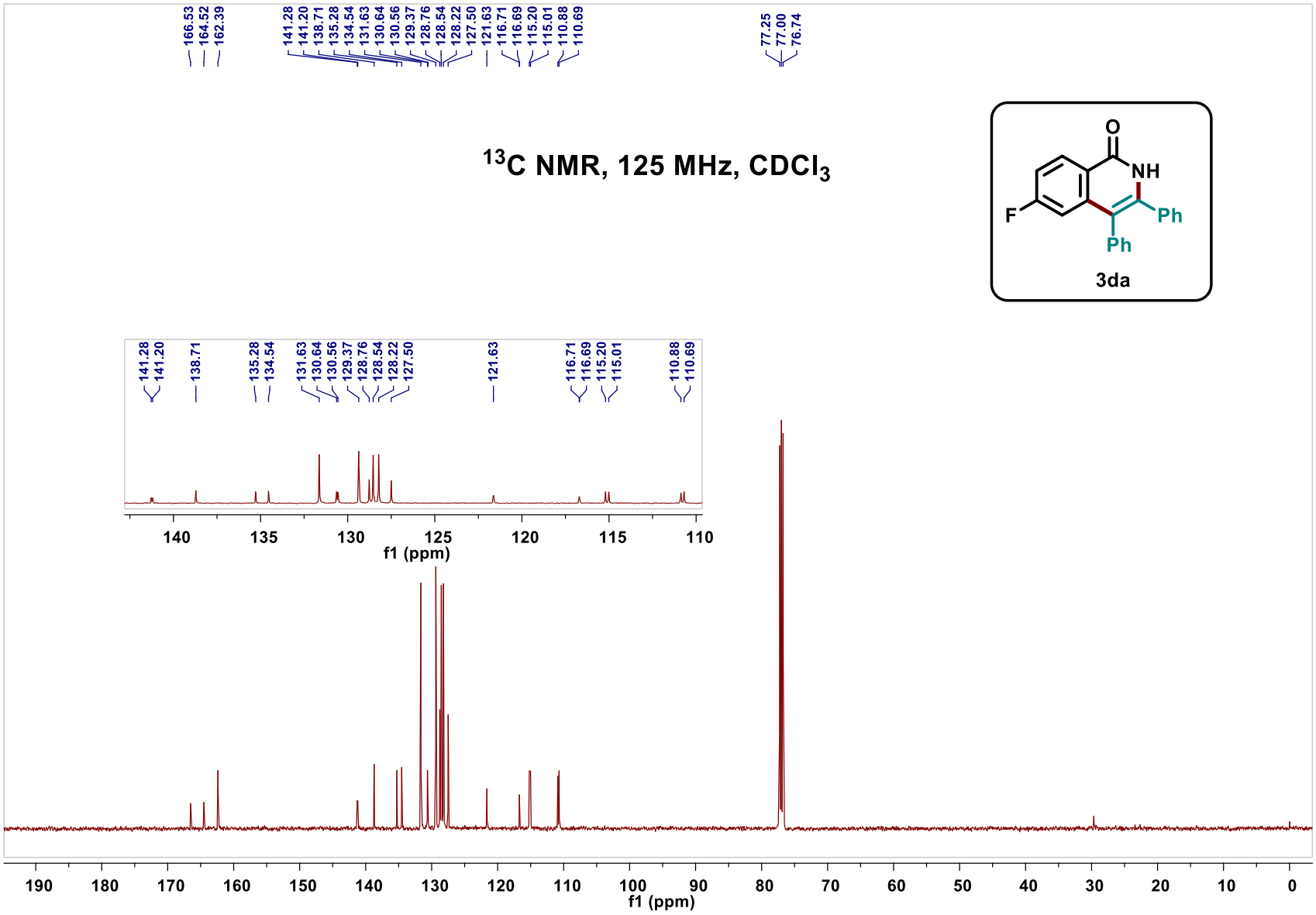
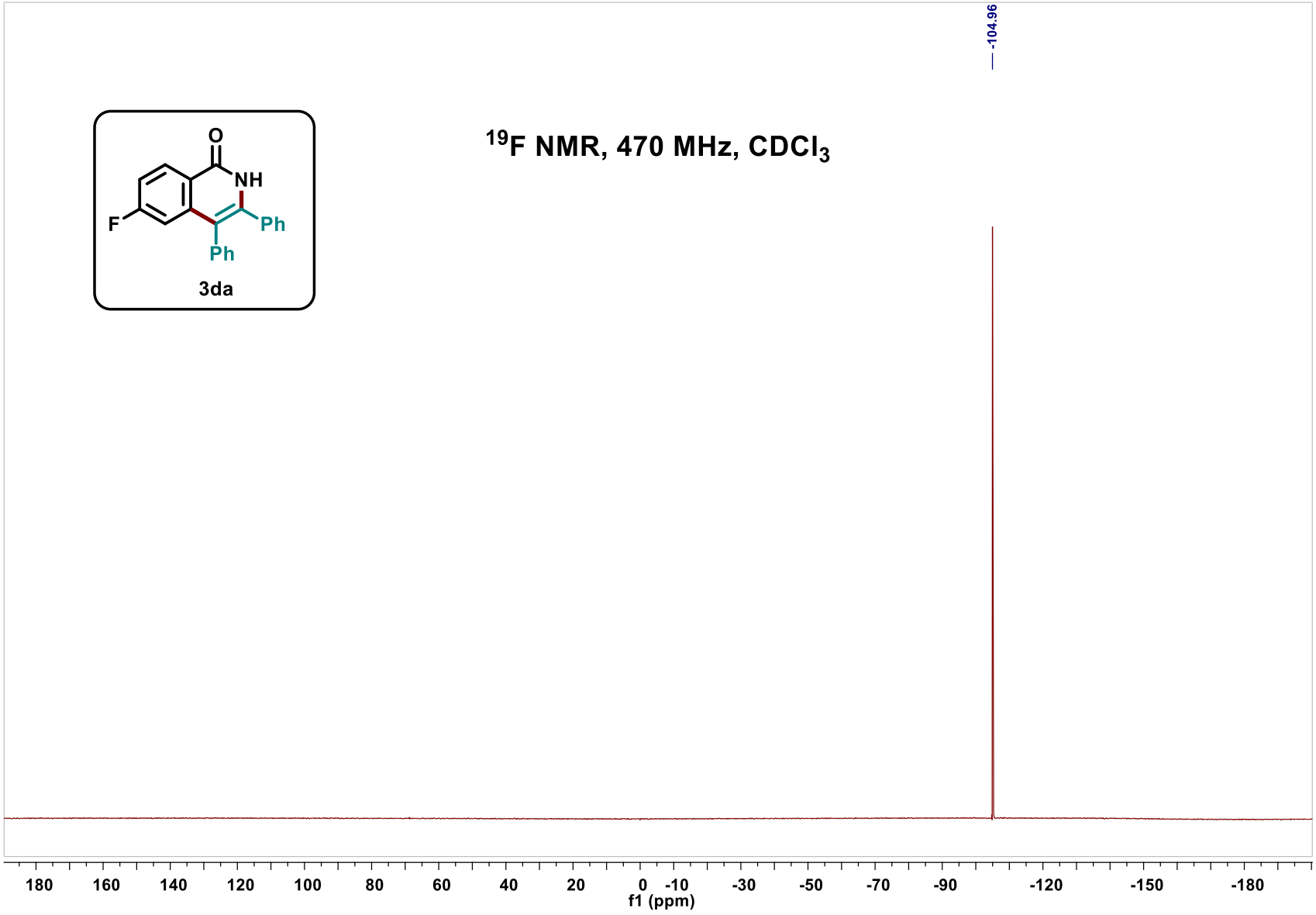
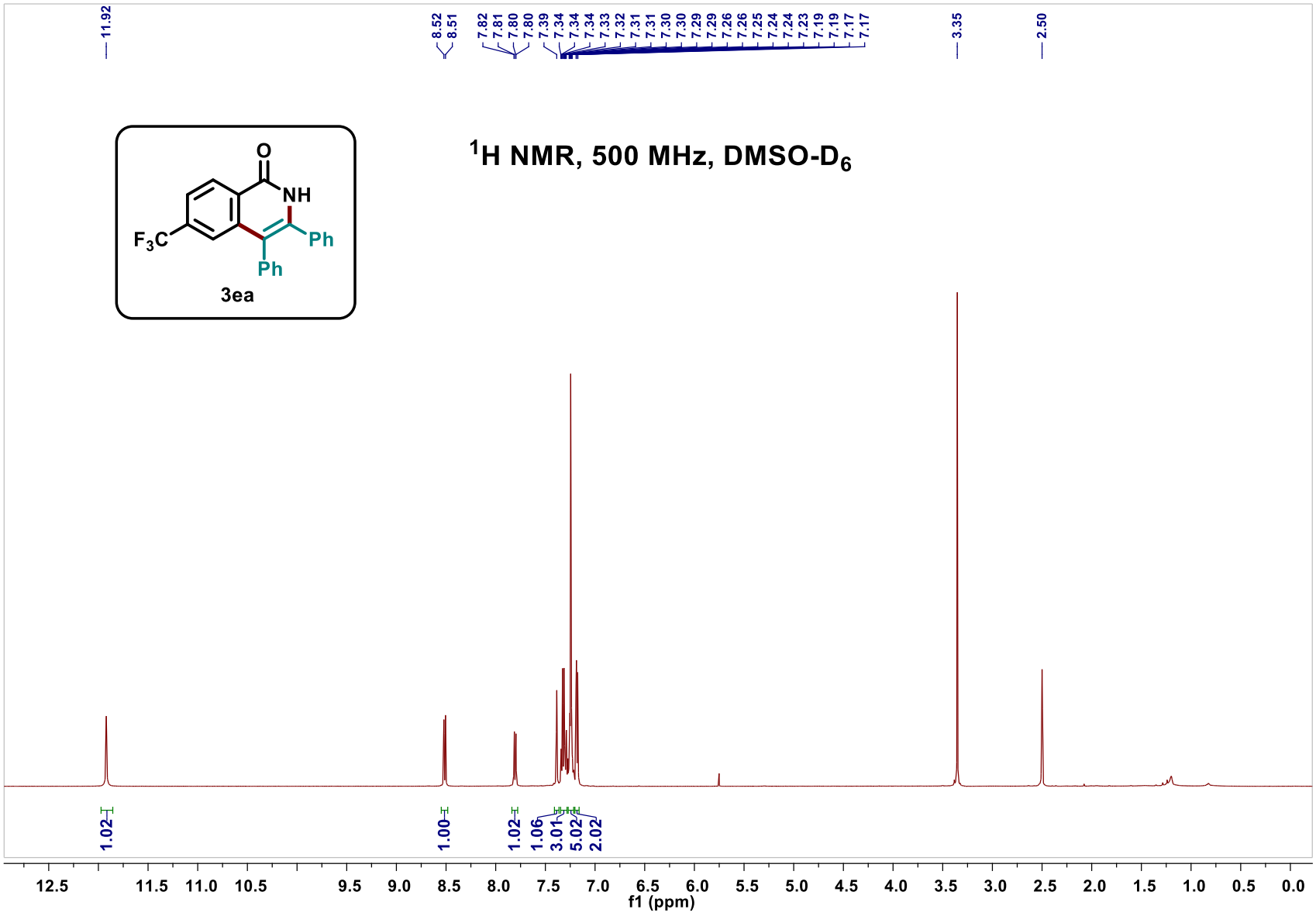
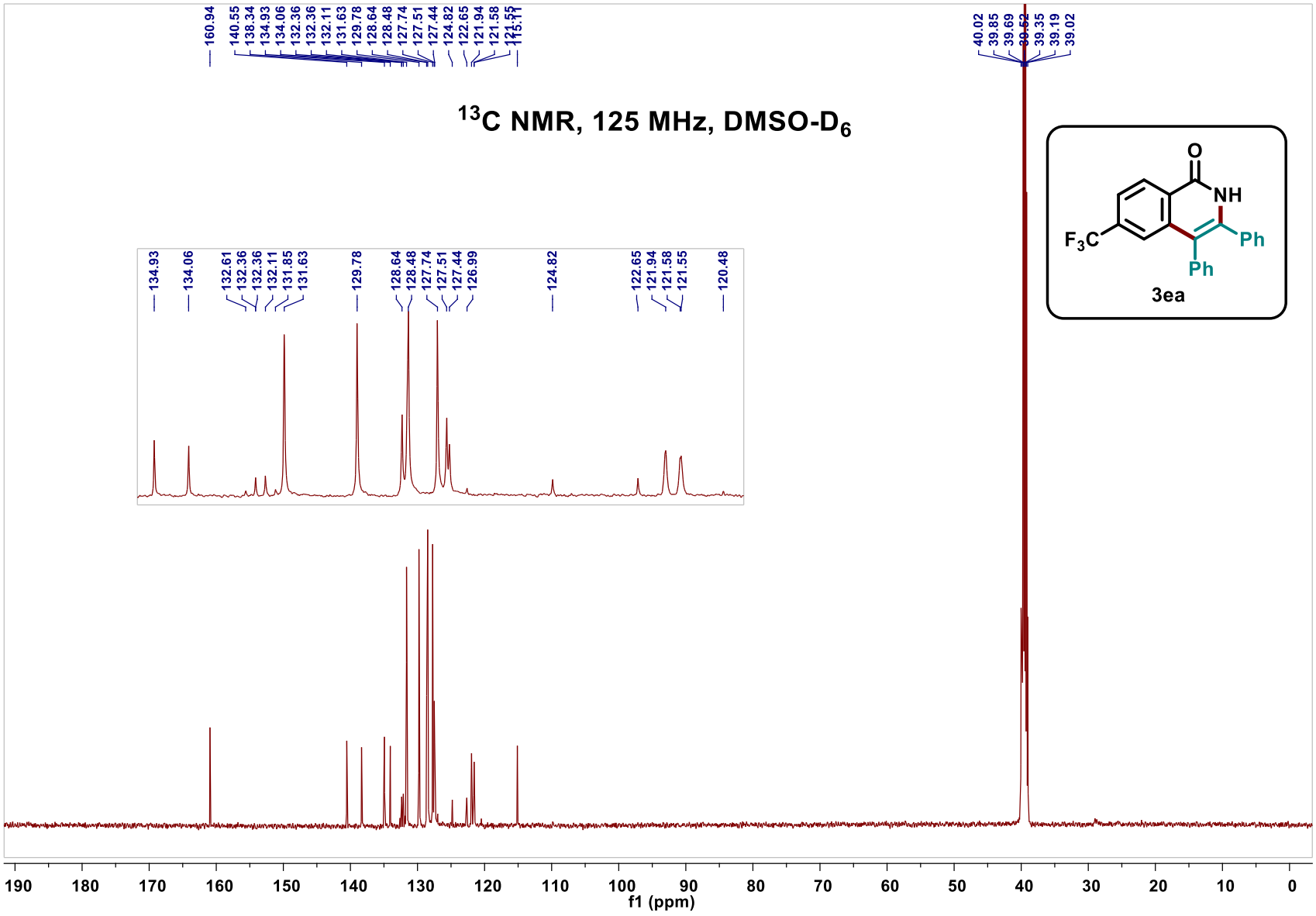
128.34
128.26
127.32
127.24
124.67
123.00
121.82
117.56
77.25
77.00
76.75
35.31
31.01
O
NH
tBu
Ph
Ph
3ca
13C NMR, 125 MHz, CDCl3
129.23
128.53
128.34
128.26
127.32
127.24
129.5
129.0
128.5
128.0
127.5
f1 (ppm)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

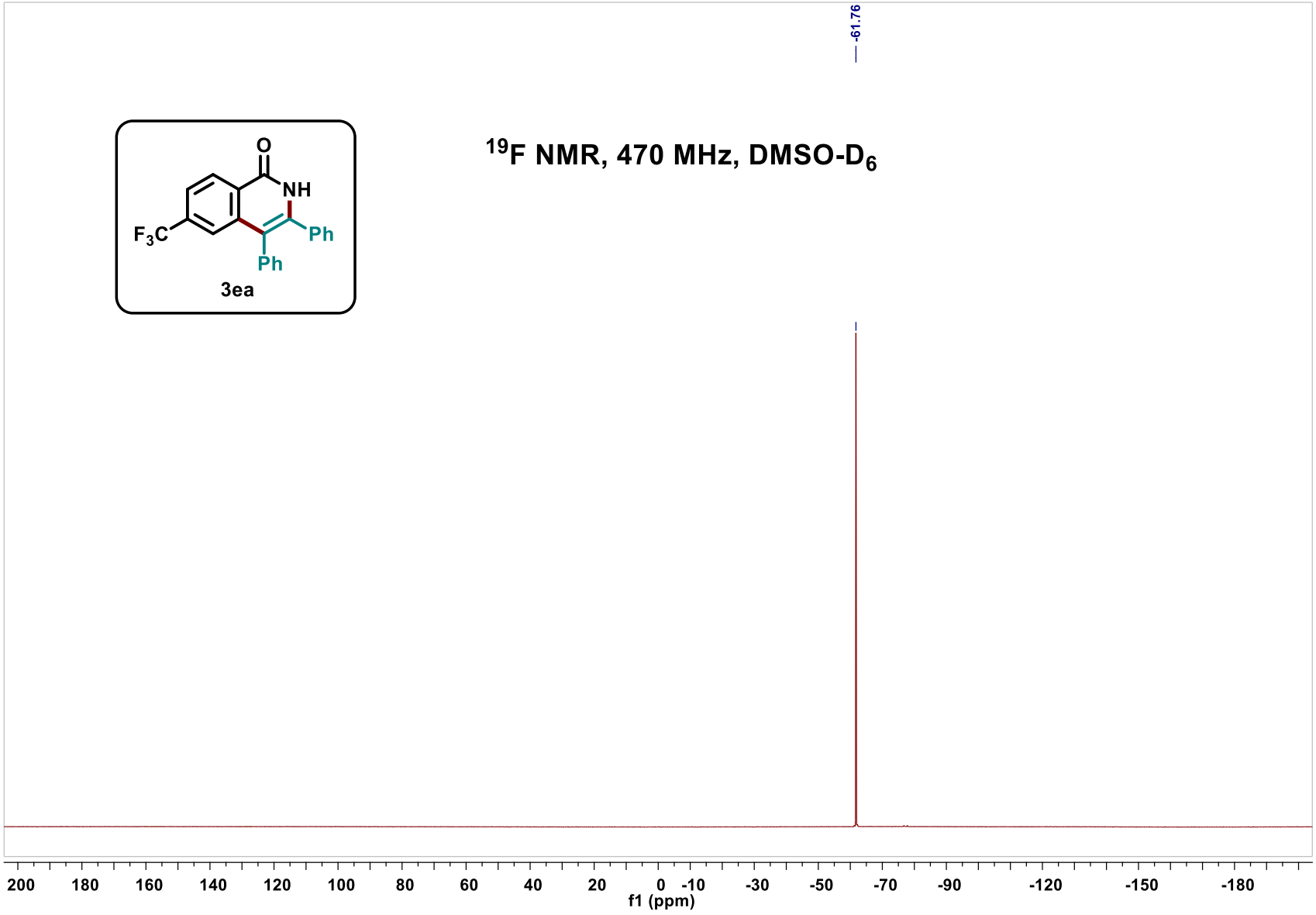

10.25
8.44
8.43
8.42
8.41
7.34
7.34
7.32
7.31
7.27
7.26
7.25
7.17
7.17
7.16
7.16
7.00
6.99
6.98
6.97
O
NH
F
Ph
Ph
3da
1H NMR, 500 MHz, CDCl3
1.00
1.00
3.06
5.06
3.05
1.00
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

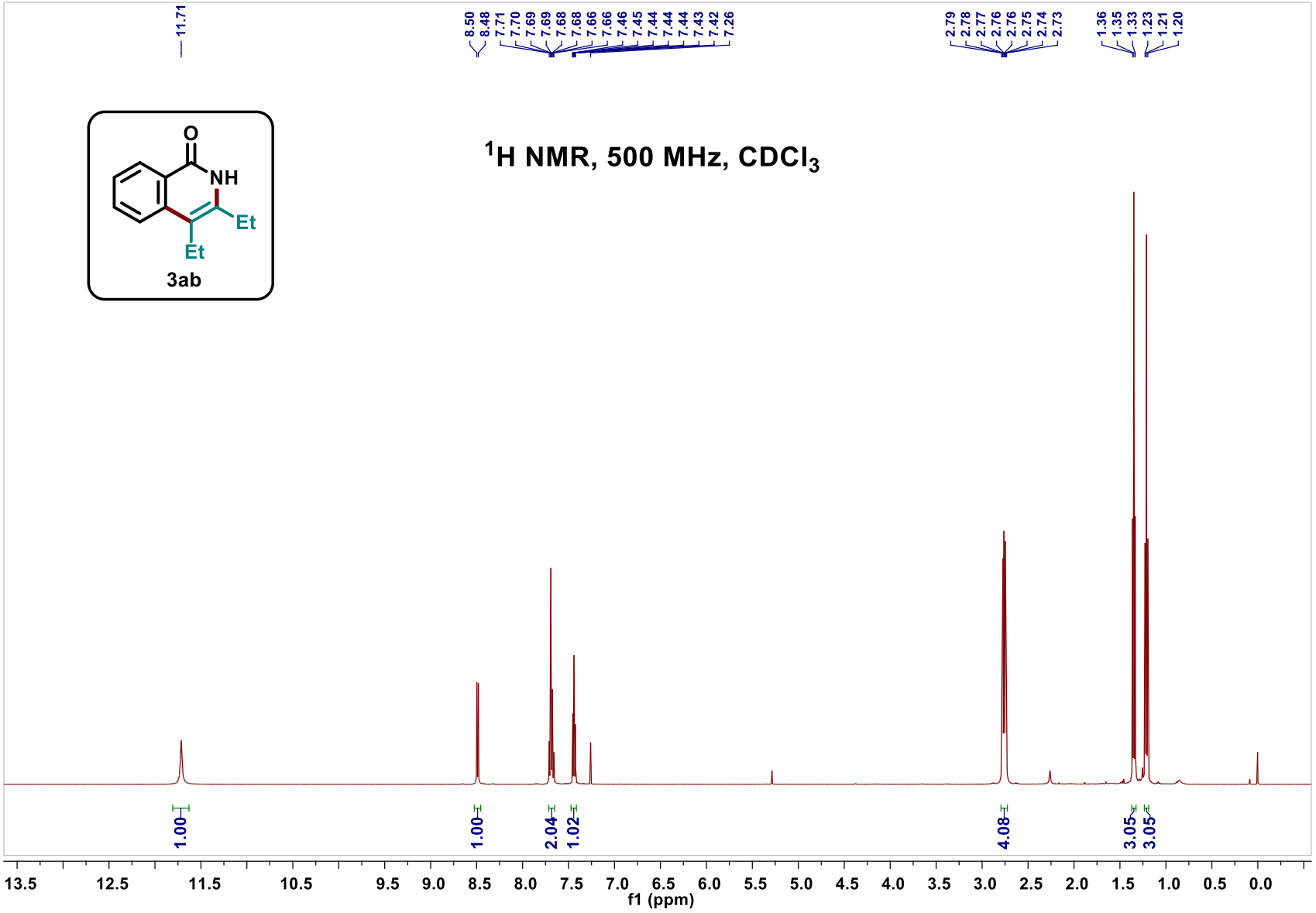

166.53
164.52
162.39
141.28
141.20
138.71
135.28
134.54
131.63
130.64
130.56
129.37
128.76
128.54
128.22
127.50
121.63
116.71
116.69
115.20
115.01
110.88
110.69
77.25
77.00
76.74
^{13}C NMR, 125 MHz, CDCl$_3$
O
NH
F
Ph
Ph
3da
f1 (ppm)
f1 (ppm)


-104.96
O
NH
F
Ph
Ph
3da
19F NMR, 470 MHz, CDCl3
180
160
140
120
100
80
60
40
20
0
-10
-30
-50
-70
-90
-120
-150
-180
f1 (ppm)


11.92
8.52
8.51
7.82
7.81
7.80
7.80
7.39
7.34
7.34
7.34
7.33
7.32
7.31
7.31
7.30
7.30
7.29
7.29
7.26
7.25
7.24
7.24
7.23
7.19
7.19
7.17
7.17
3.35
2.50
O
NH
F3C
Ph
Ph
3ea
1H NMR, 500 MHz, DMSO-D6
1.02
1.00
1.02
1.06
3.01
5.02
2.02
12.5
11.5
11.0
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


160.94
140.55
138.34
134.93
134.06
132.36
132.36
132.11
131.63
129.78
128.64
128.48
127.74
127.51
127.44
124.82
122.65
121.94
121.58
121.55
120.48
40.02
39.85
39.69
39.52
39.35
39.19
39.02
13C NMR, 125 MHz, DMSO-D6
O
NH
F3C
Ph
Ph
3ea
f1 (ppm)


-61.76
O
NH
F3C
Ph
Ph
3ea
19F NMR, 470 MHz, DMSO-D6
200
180
160
140
120
100
80
60
40
20
0
-10
-30
-50
-70
-90
-120
-150
-180
f1 (ppm)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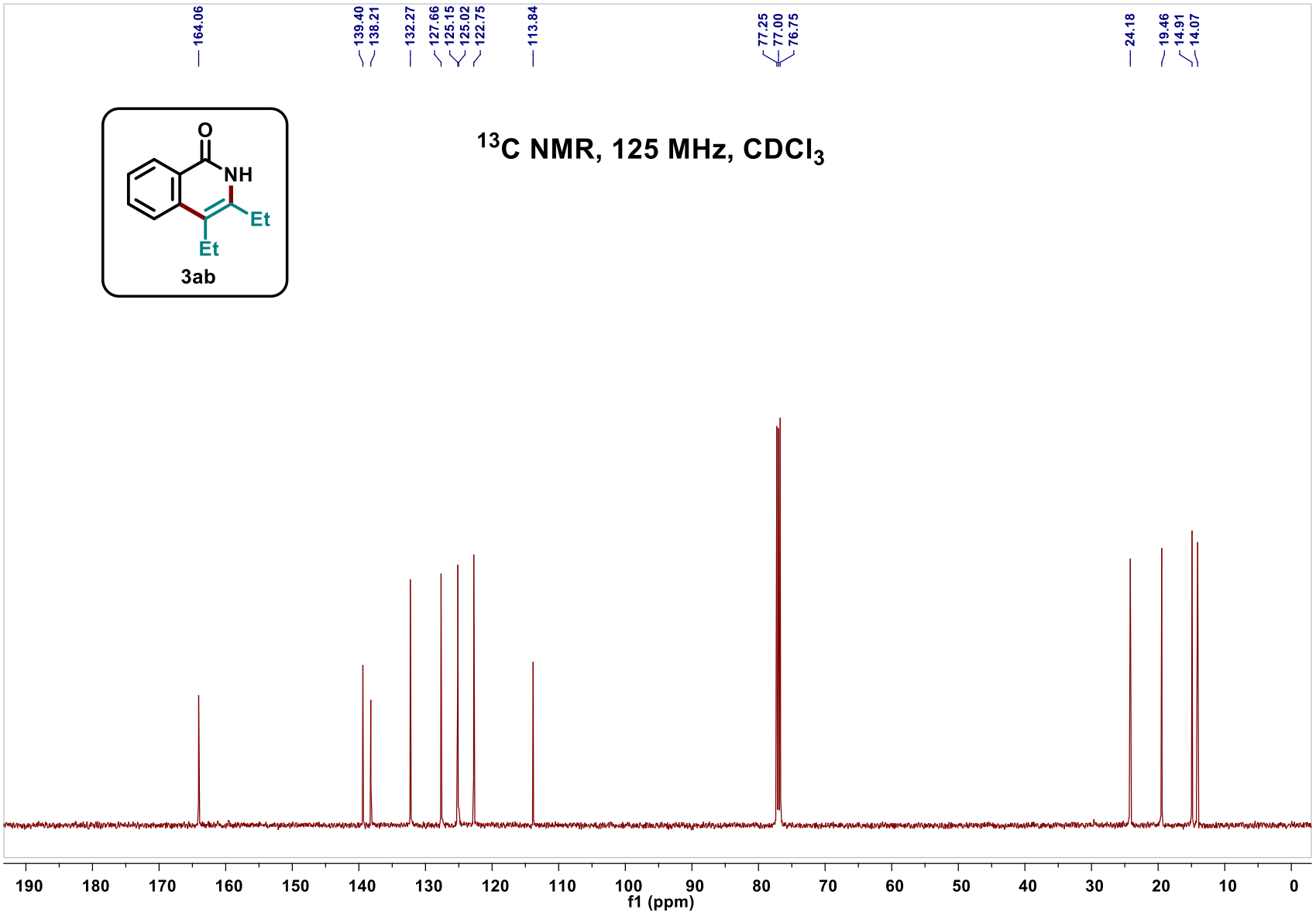
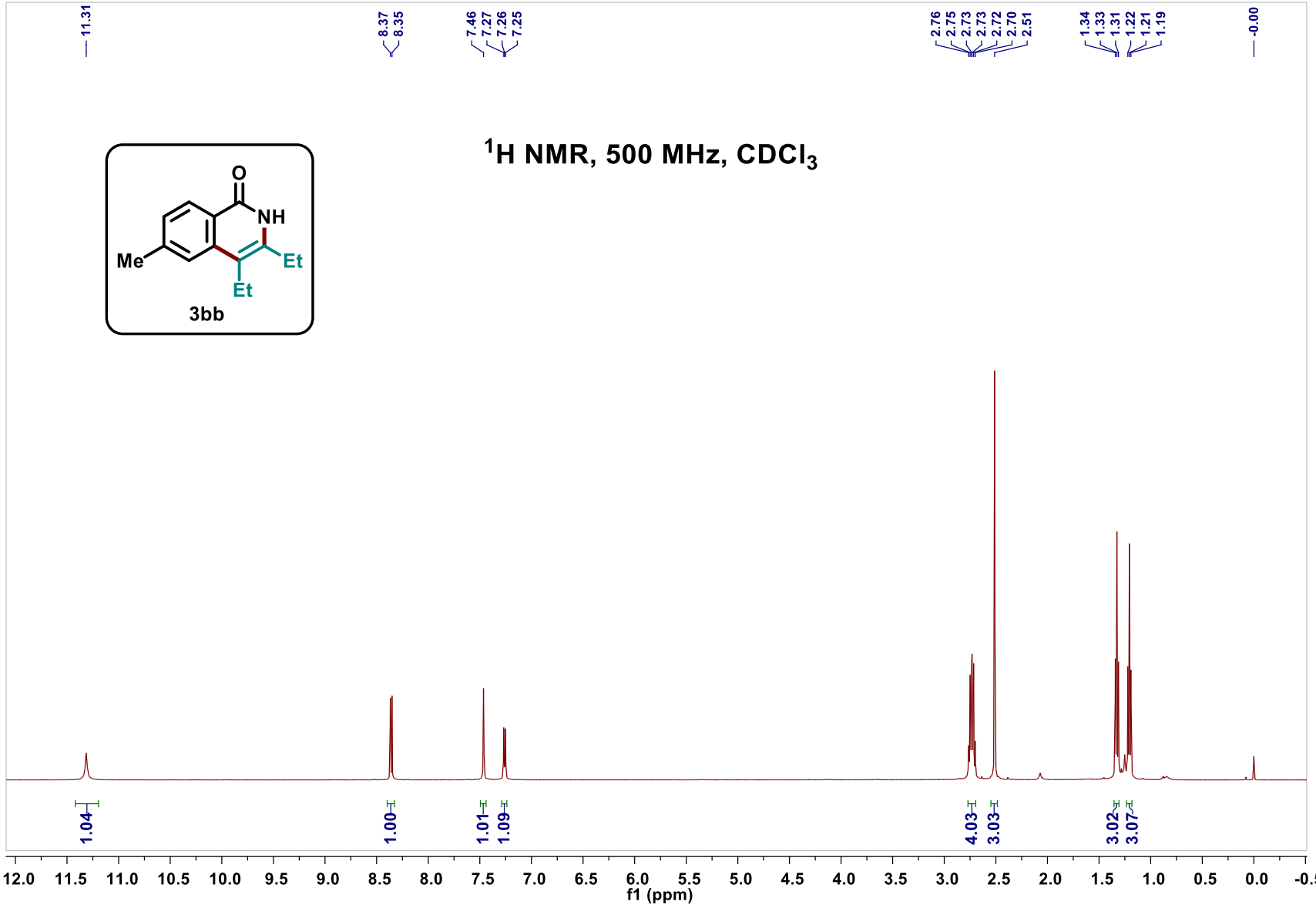
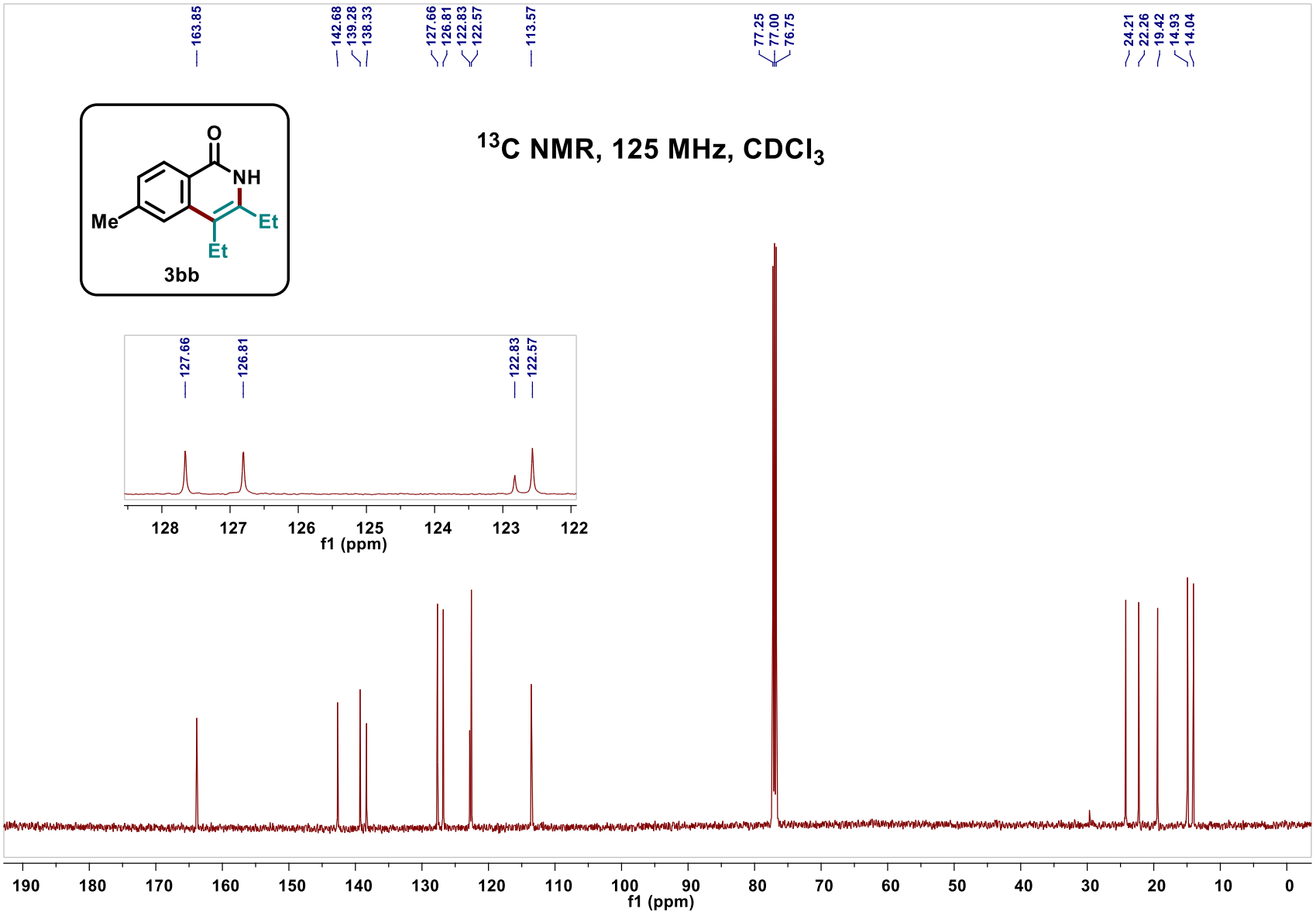
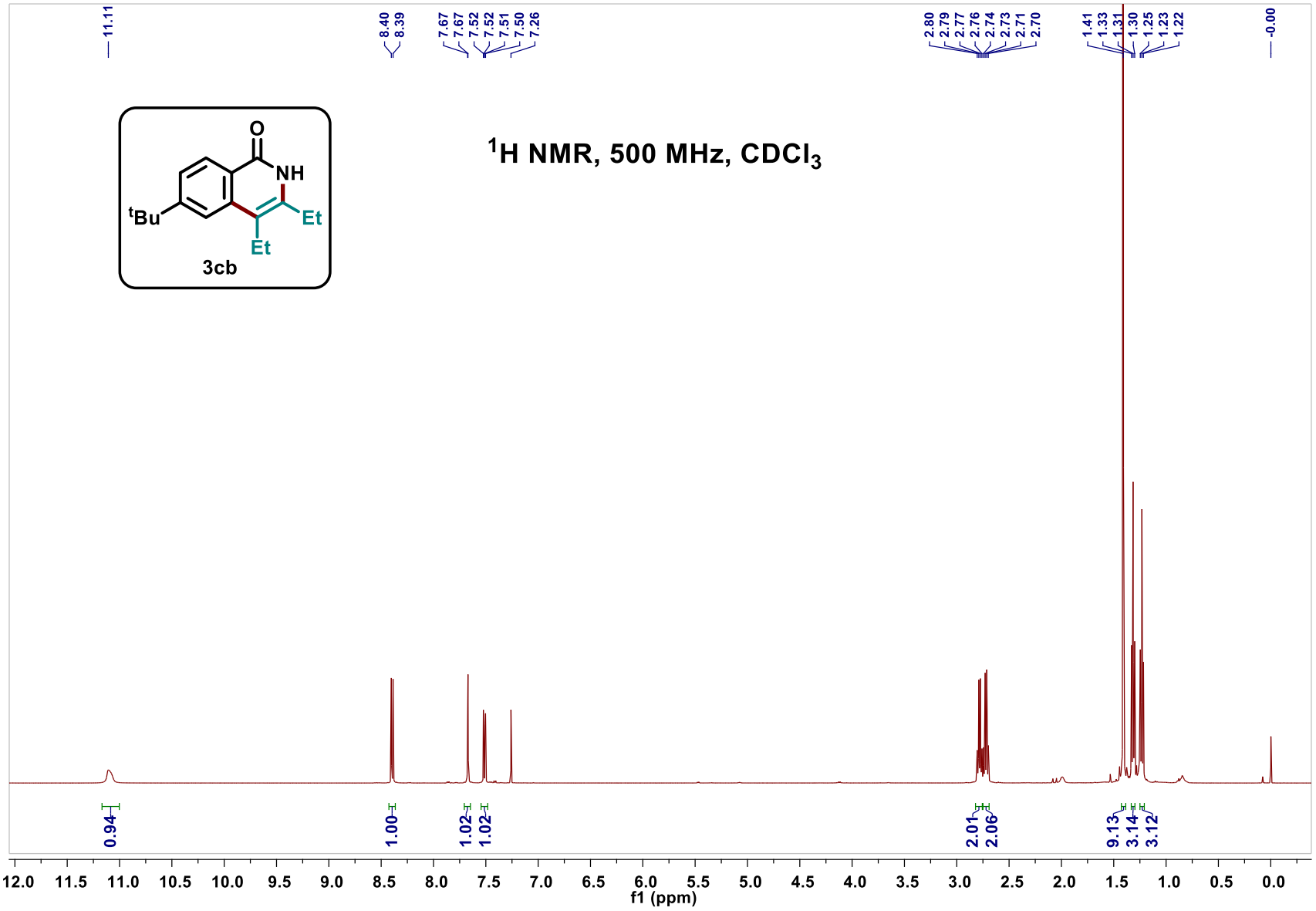
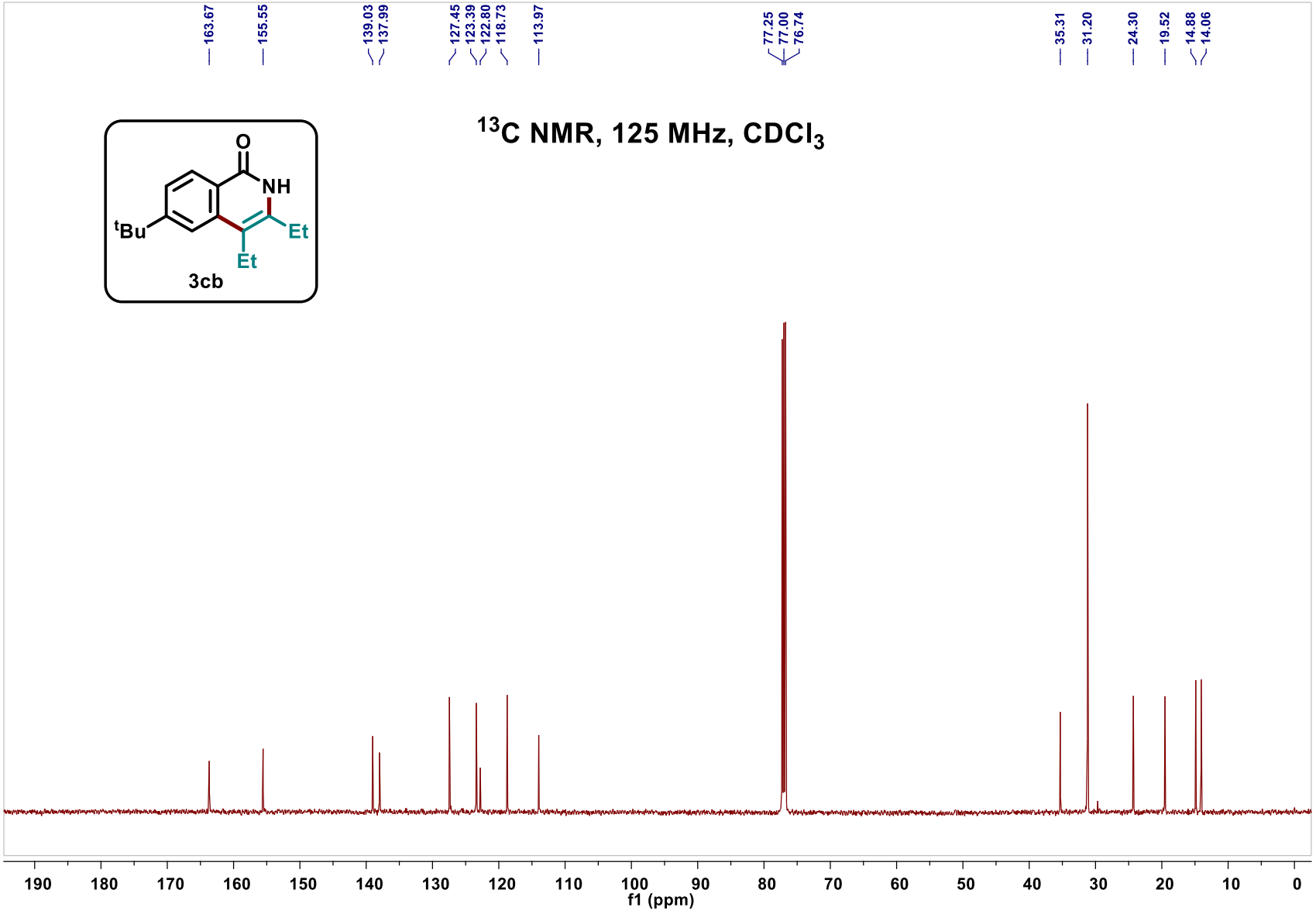
8.50
8.48
7.71
7.70
7.69
7.69
7.68
7.68
7.66
7.66
7.46
7.45
7.44
7.44
7.44
7.43
7.42
7.26
2.79
2.78
2.77
2.76
2.76
2.75
2.74
2.73
1.36
1.35
1.33
1.23
1.21
1.20
O
NH
Et
Et
3ab
1H NMR, 500 MHz, CDCl3
1.00
1.00
2.04
1.02
4.08
3.05
3.05
13.5
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

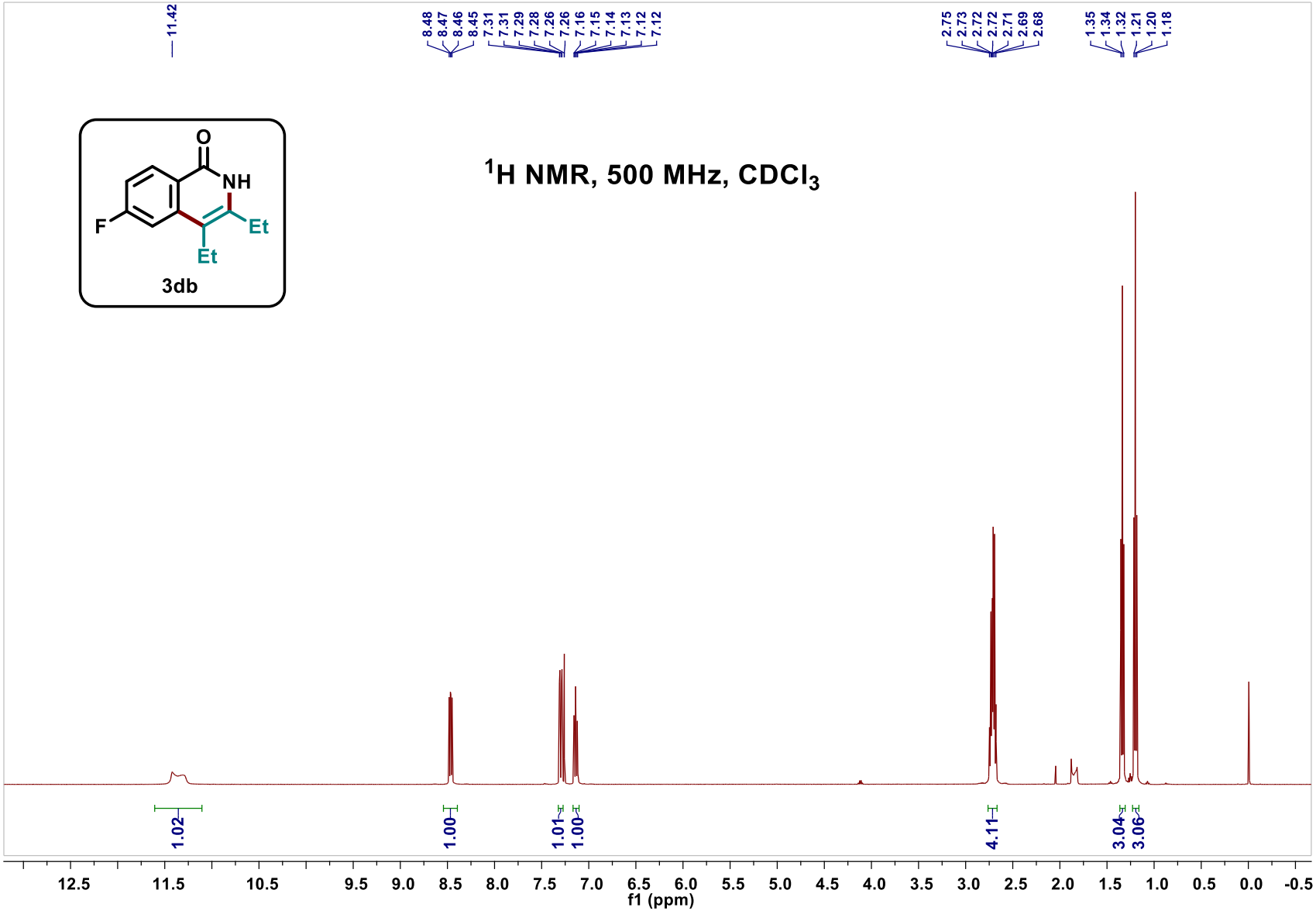

164.06
139.40
138.21
132.27
127.66
125.15
125.02
122.75
113.84
77.25
77.00
76.75
24.18
19.46
14.91
14.07
O
NH
Et
Et
3ab
13C NMR, 125 MHz, CDCl3
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


11.31
8.37
8.35
7.46
7.27
7.26
7.25
2.76
2.75
2.73
2.73
2.72
2.70
2.51
1.34
1.33
1.31
1.22
1.21
1.19
-0.00
1H NMR, 500 MHz, CDCl3
O
NH
Me
Et
Et
3bb
1.04
1.00
1.01
1.09
4.03
3.03
3.02
3.07
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 0.0
f1 (ppm)


163.85
142.68
139.28
138.33
127.66
126.81
122.83
122.57
113.57
77.25
77.00
76.75
24.21
22.26
19.42
14.93
14.04
O
NH
Me
Et
Et
3bb
13C NMR, 125 MHz, CDCl3
127.66
126.81
122.83
122.57
128
127
126
125
124
123
122
f1 (ppm)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


11.11
8.40
8.39
7.67
7.67
7.52
7.52
7.51
7.50
7.26
2.80
2.79
2.77
2.76
2.74
2.73
2.71
2.70
1.41
1.33
1.31
1.30
1.25
1.23
1.22
-0.00
O
NH
tBu
Et
Et
3cb
1H NMR, 500 MHz, CDCl3
0.94
1.00
1.02
1.02
2.01
2.06
9.13
3.14
3.12
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 0.0
f1 (ppm)


163.67
155.55
139.03
137.99
127.45
123.39
122.80
118.73
113.97
77.25
77.00
76.74
35.31
31.20
24.30
19.52
14.88
14.06
13C NMR, 125 MHz, CDCl3
O
NH
tBu
Et
Et
3cb
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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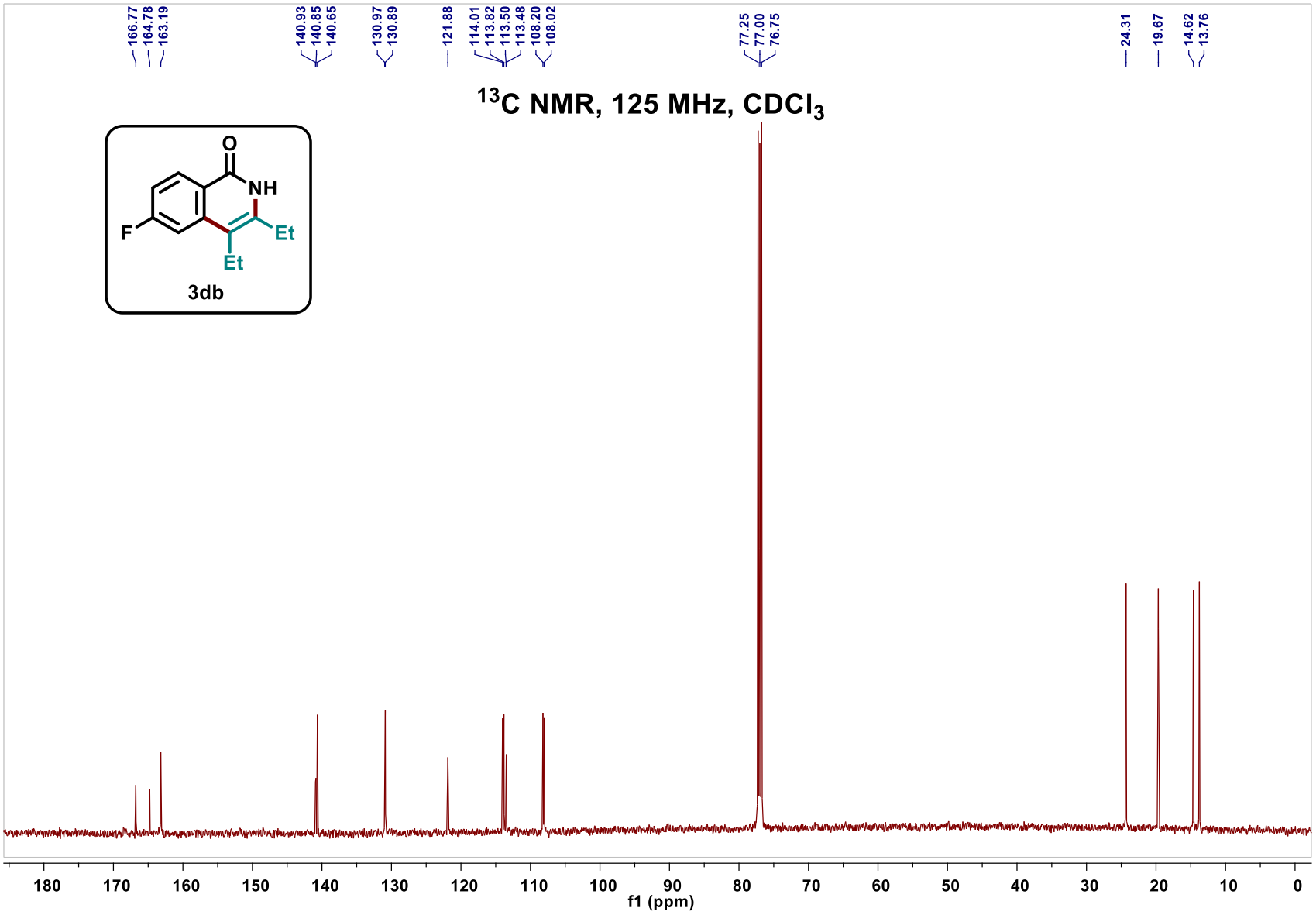
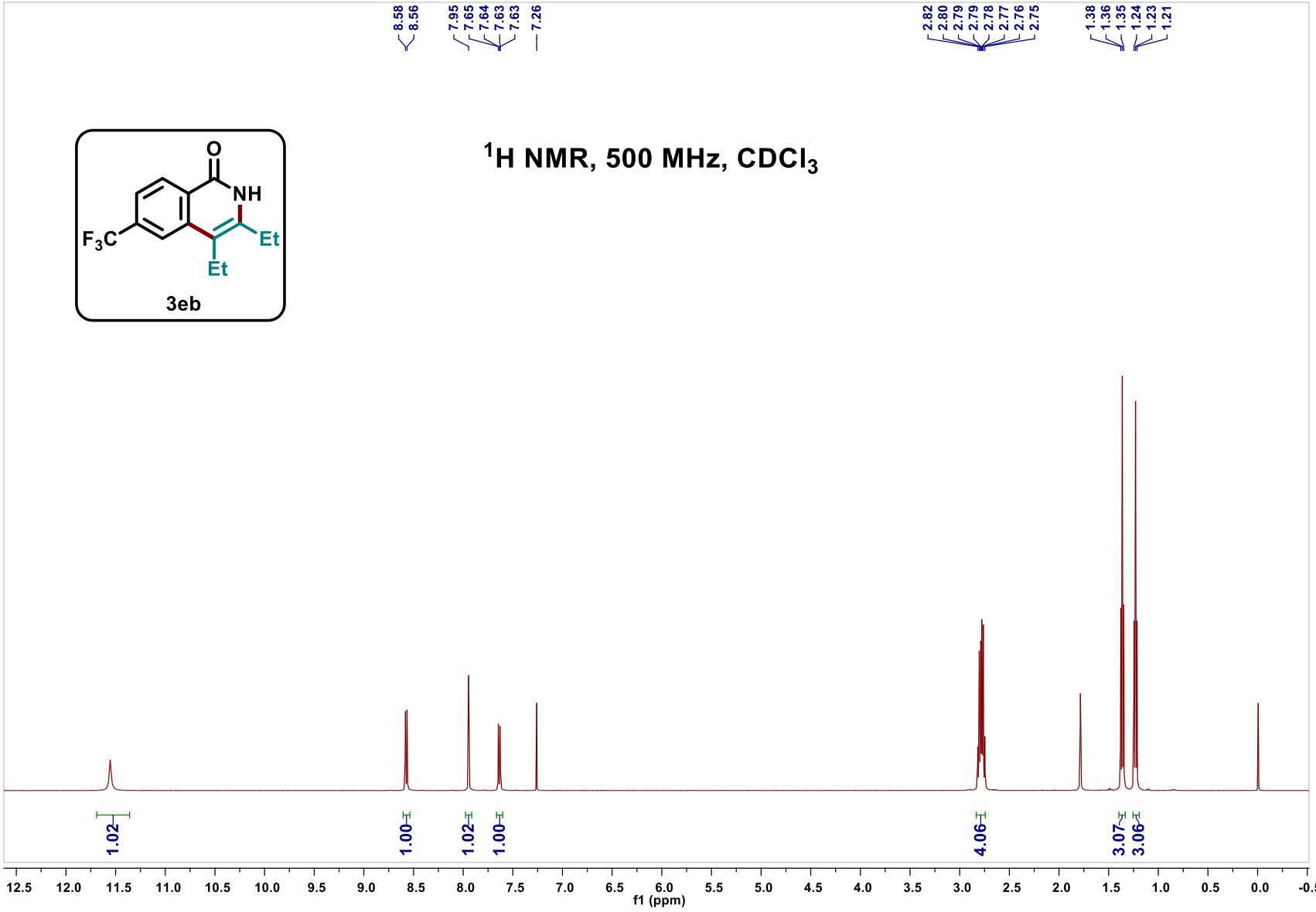
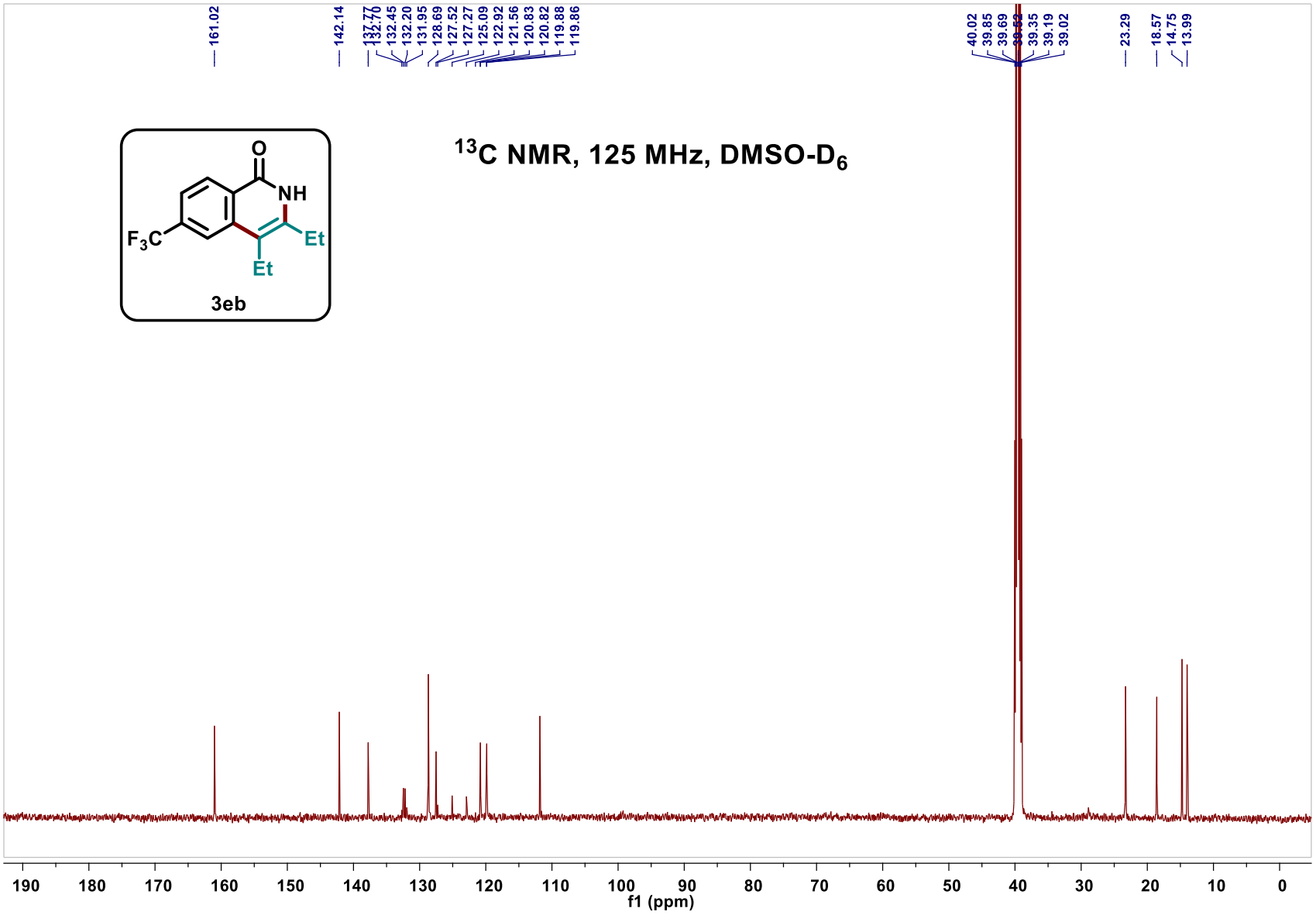
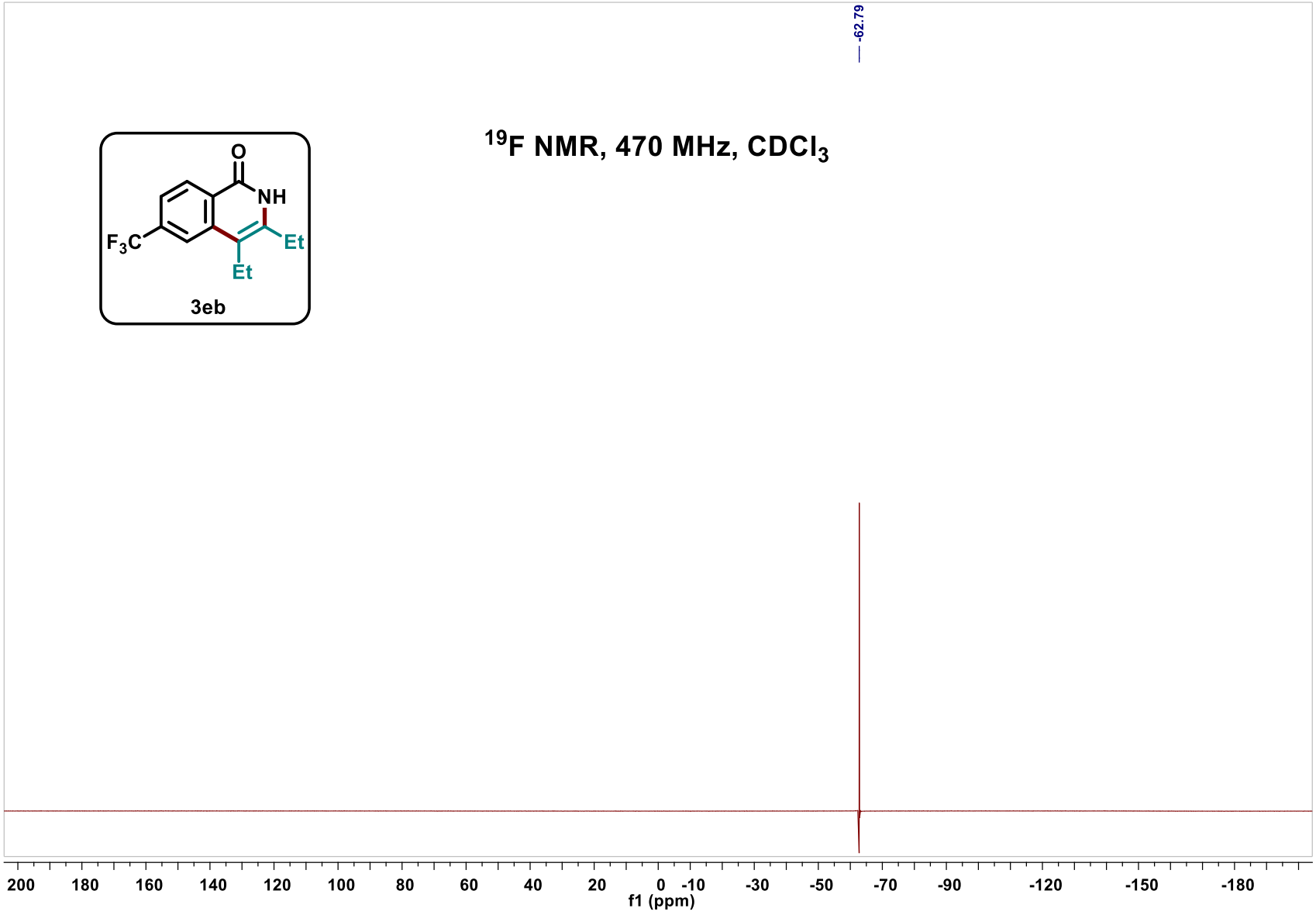
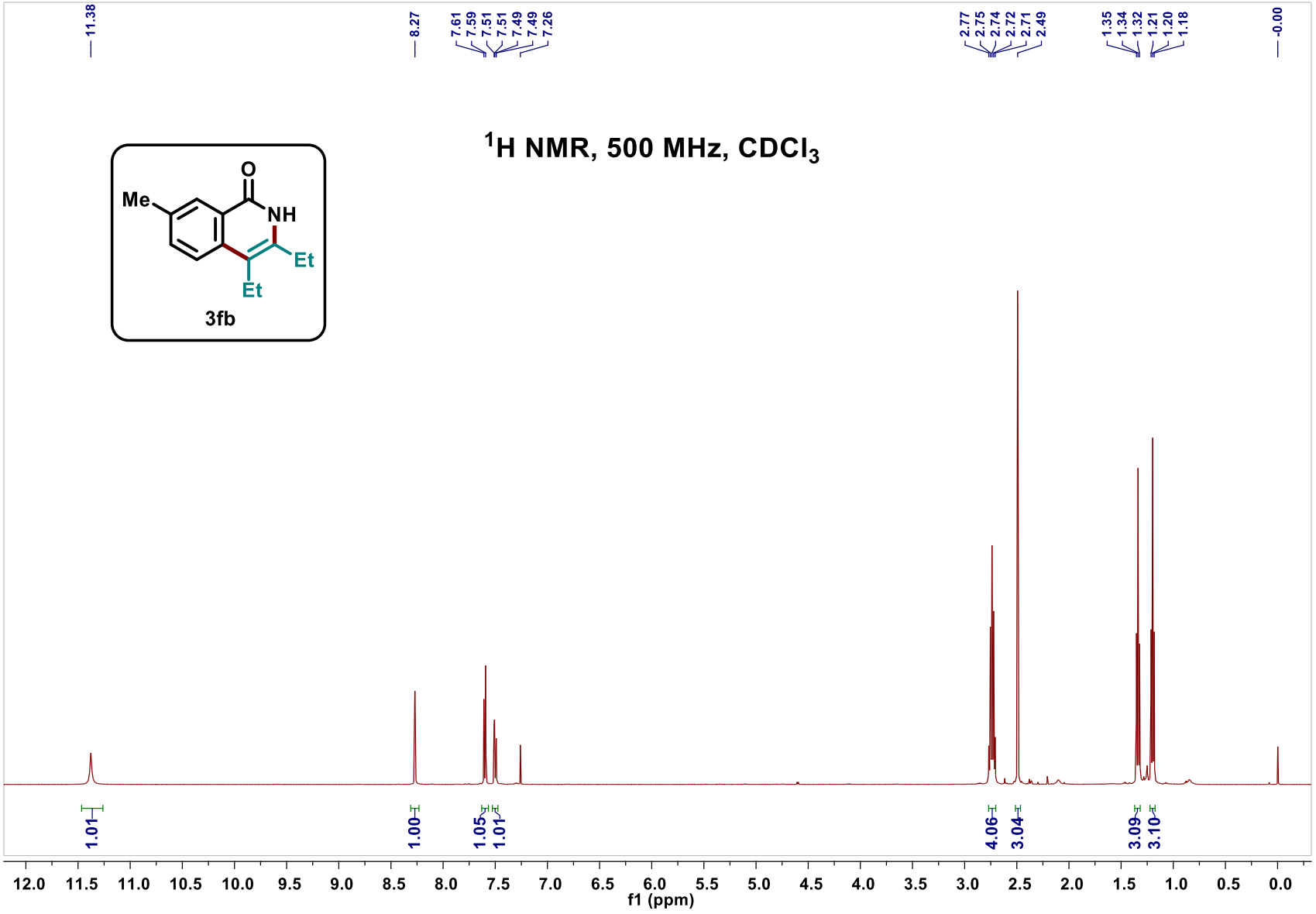
8.48
8.47
8.46
8.45
7.31
7.29
7.28
7.26
7.16
7.15
7.14
7.13
7.12
7.12
2.75
2.73
2.72
2.72
2.71
2.69
2.68
1.35
1.34
1.32
1.21
1.20
1.18
O
NH
F
Et
Et
3db
1H NMR, 500 MHz, CDCl3
1.02
1.00
1.01
1.00
4.11
3.04
3.06
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

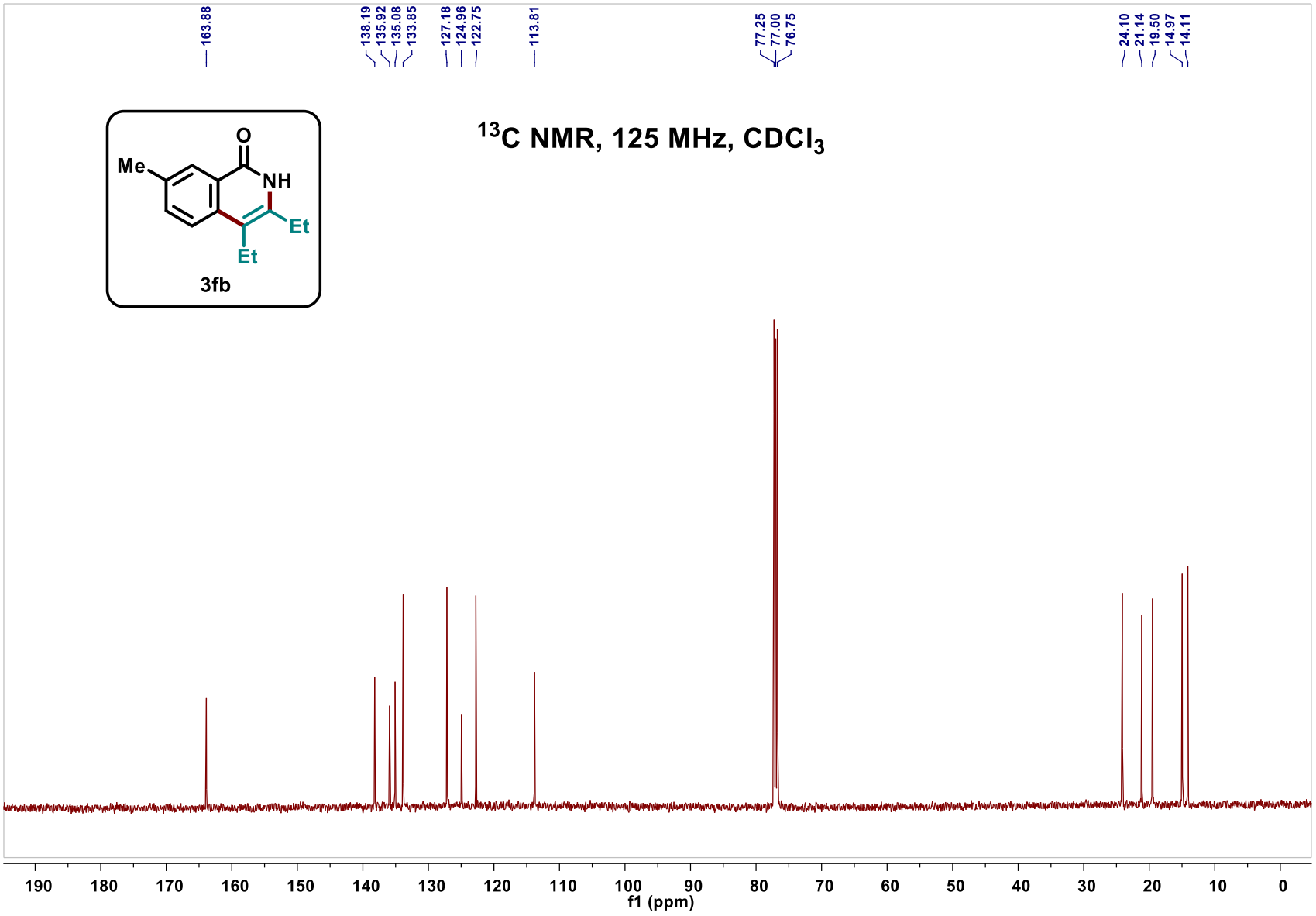

166.77
164.78
163.19
140.93
140.85
140.65
130.97
130.89
121.88
114.01
113.82
113.50
113.48
108.20
108.02
77.25
77.00
76.75
24.31
19.67
14.62
13.76
13C NMR, 125 MHz, CDCl3
O
NH
F
Et
Et
3db
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.58
8.56
7.95
7.65
7.64
7.63
7.63
7.26
2.82
2.80
2.79
2.79
2.78
2.77
2.76
2.75
1.38
1.36
1.35
1.24
1.23
1.21
O
NH
F3C
Et
Et
3eb
1H NMR, 500 MHz, CDCl3
1.02
1.00
1.02
1.00
4.06
3.07
3.06
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 0.0 -0.5
f1 (ppm)


161.02
142.14
137.77
132.70
132.45
132.20
131.95
128.69
127.52
127.27
125.09
122.92
121.56
120.83
120.82
119.88
119.86
40.02
39.85
39.69
39.52
39.35
39.19
39.02
23.29
18.57
14.75
13.99
O
NH
F3C
Et
Et
3eb
13C NMR, 125 MHz, DMSO-D6
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


-62.79
O
NH
F3C
Et
Et
3eb
19F NMR, 470 MHz, CDCl3
200
180
160
140
120
100
80
60
40
20
0
-10
-30
-50
-70
-90
-120
-150
-180
f1 (ppm)


11.38
8.27
7.61
7.59
7.51
7.51
7.49
7.49
7.26
2.77
2.75
2.74
2.72
2.71
2.49
1.35
1.34
1.32
1.21
1.20
1.18
-0.00
1H NMR, 500 MHz, CDCl3
O
Me
NH
Et
Et
3fb
1.01
1.00
1.05
1.01
4.06
3.04
3.09
3.10
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 0.0
f1 (ppm)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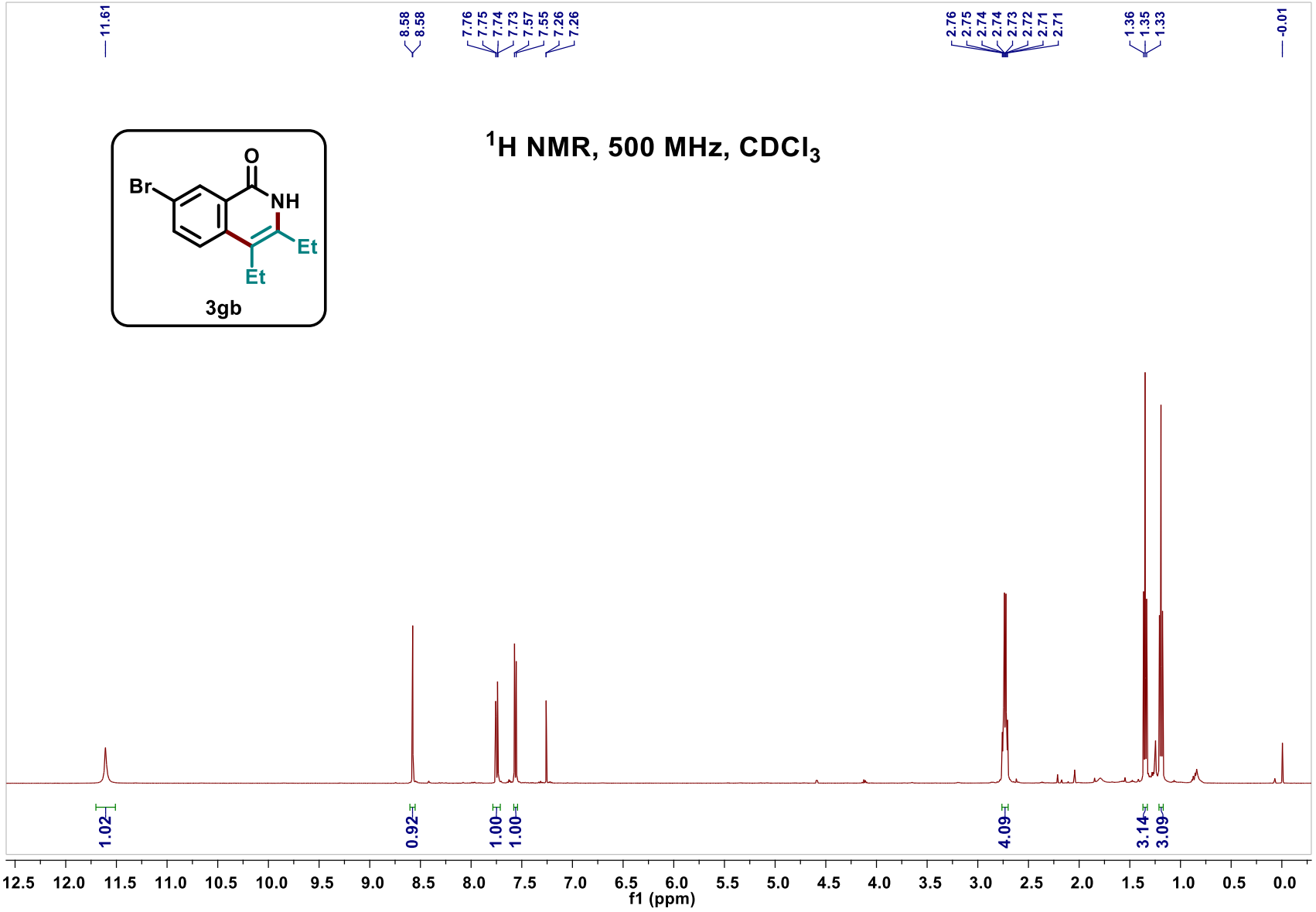
138.19
135.92
135.08
133.85
127.18
124.96
122.75
113.81
77.25
77.00
76.75
24.10
21.14
19.50
14.97
14.11
O
NH
Me
Et
Et
3fb
13C NMR, 125 MHz, CDCl3
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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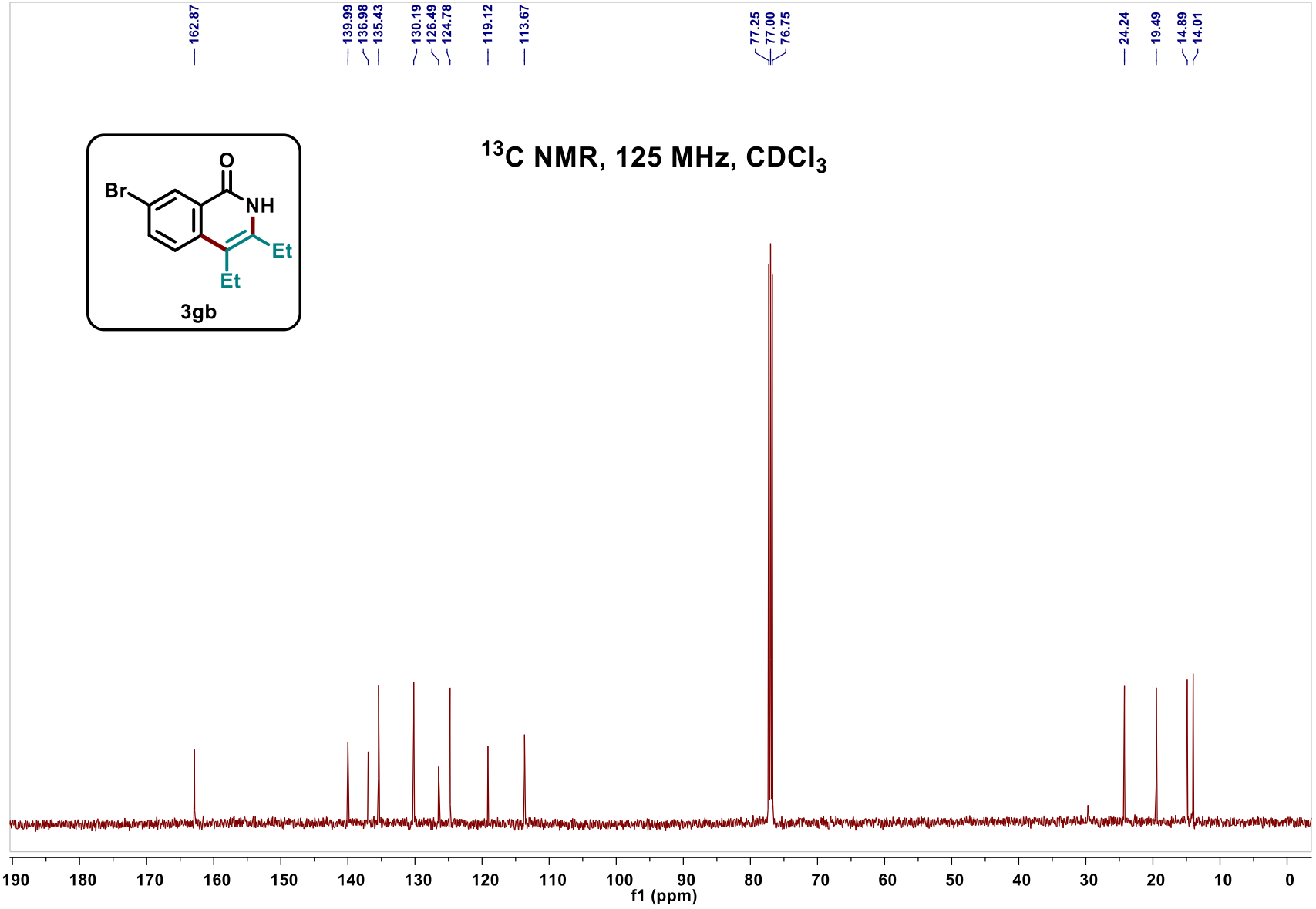
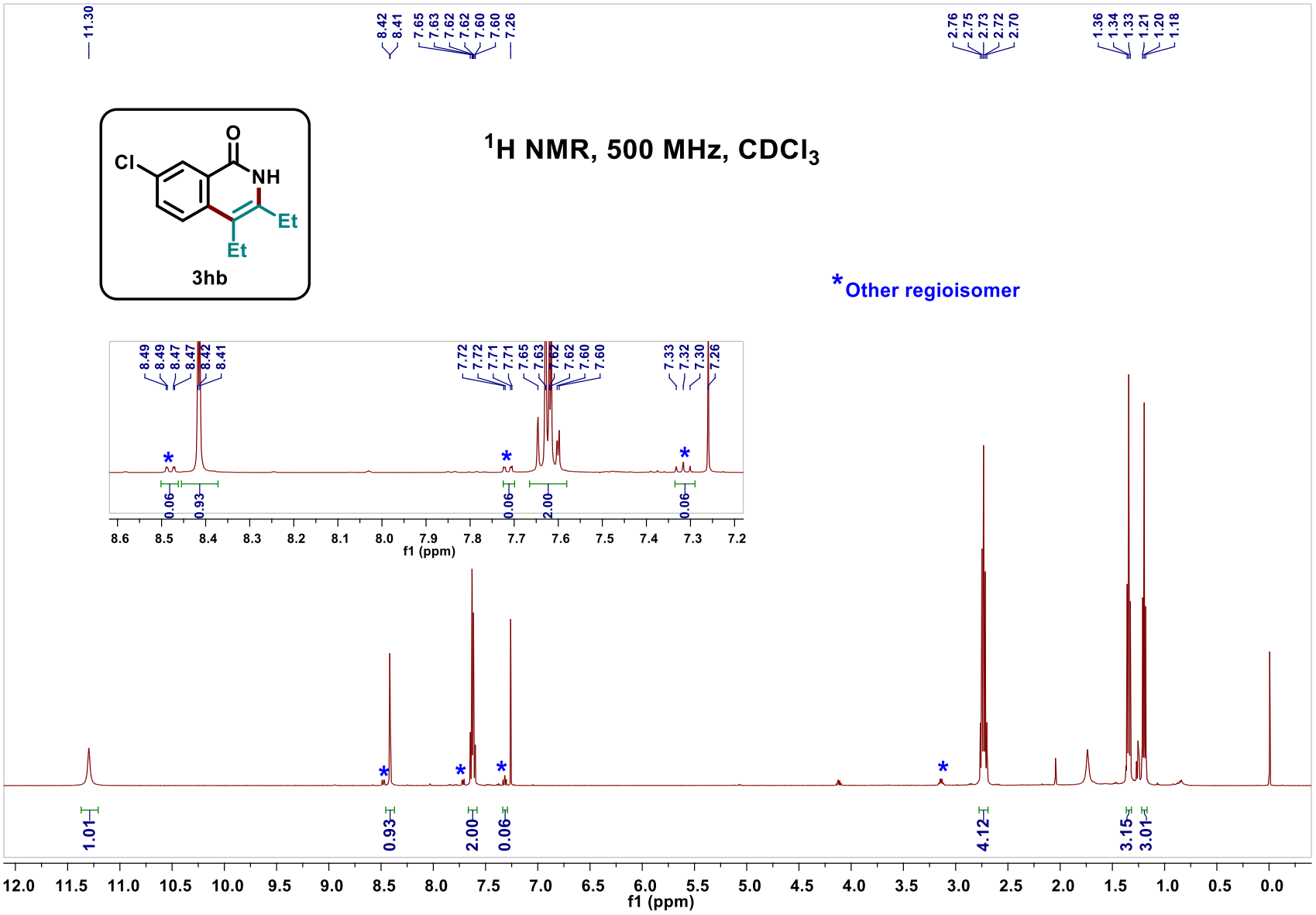
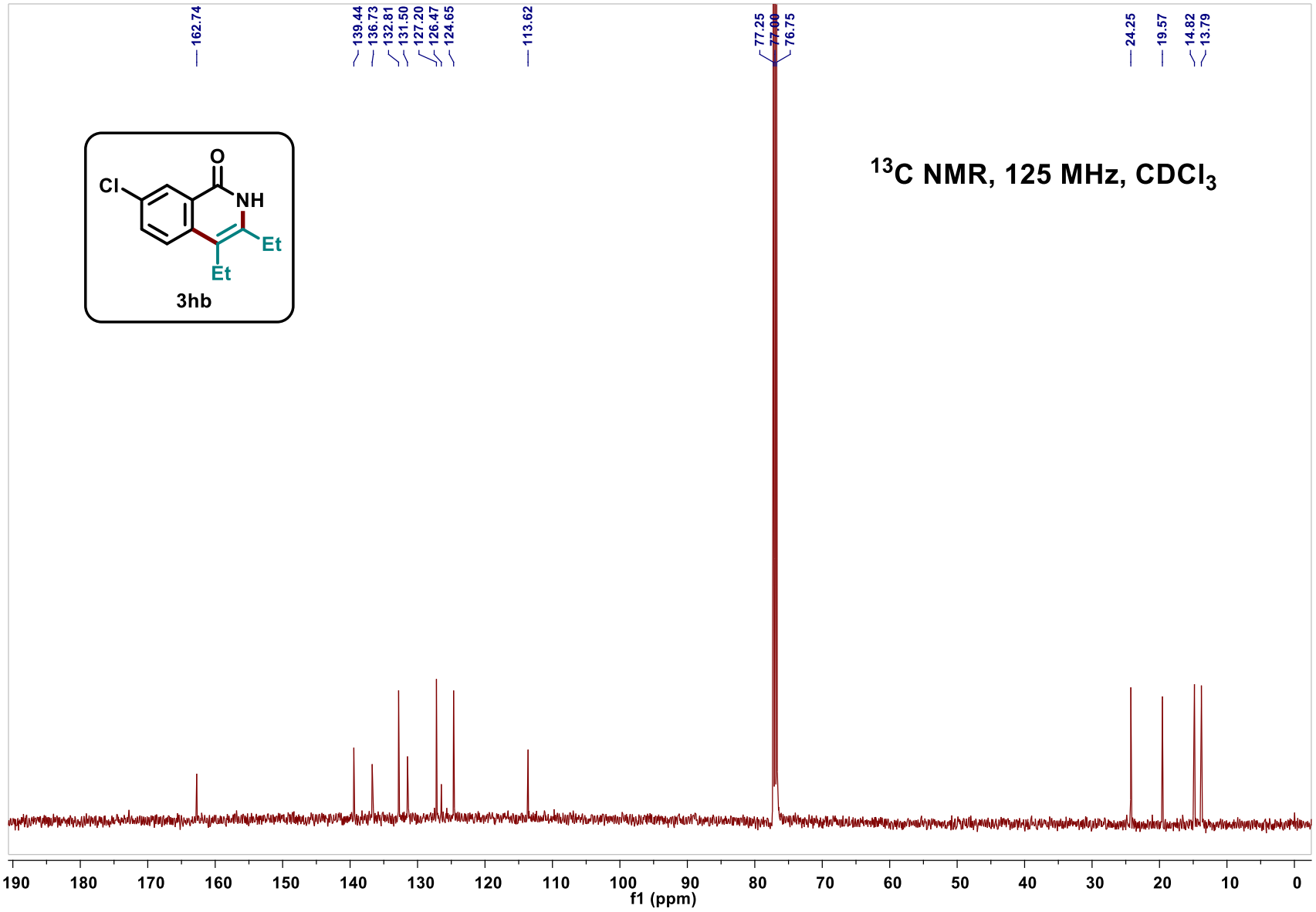
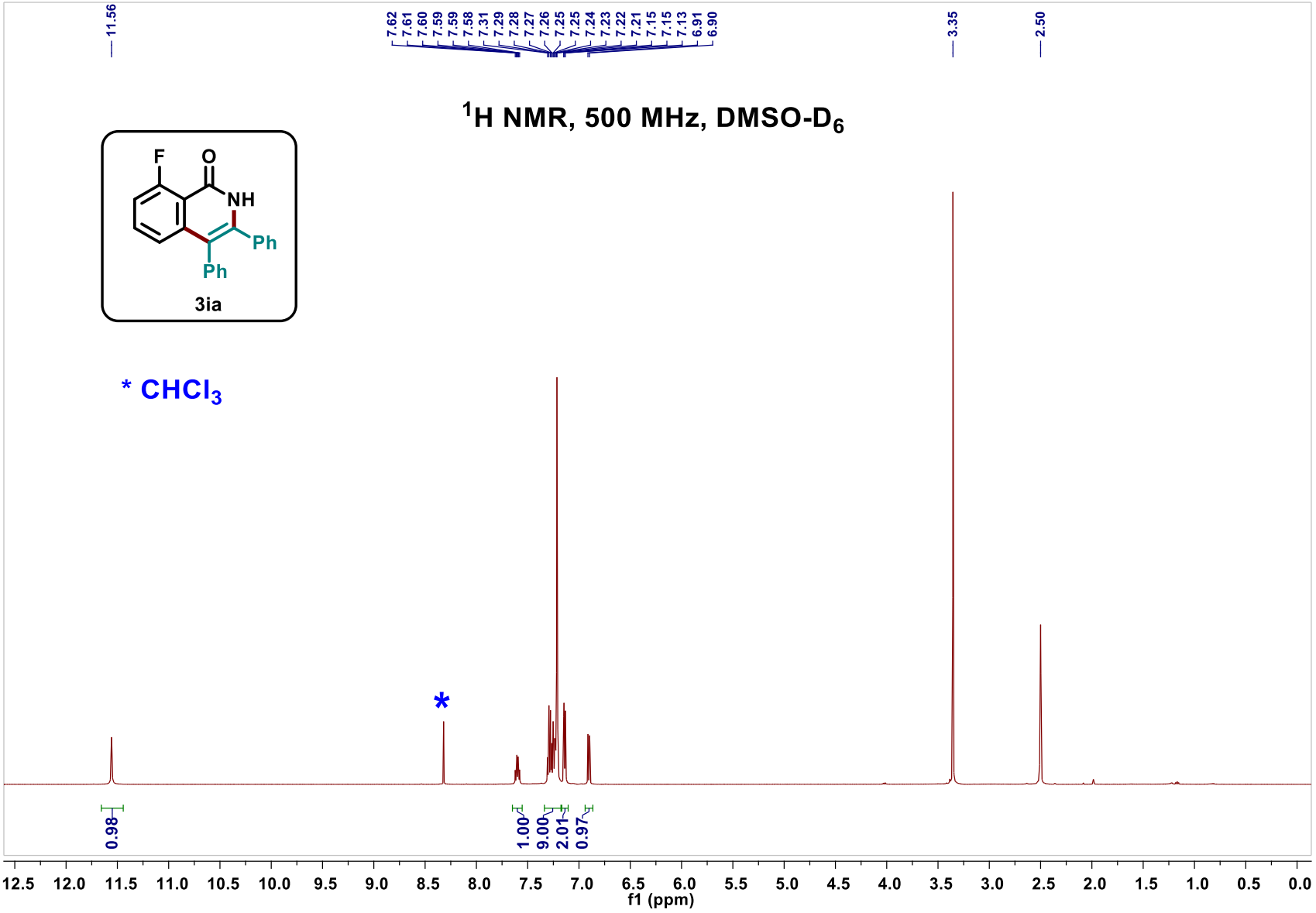
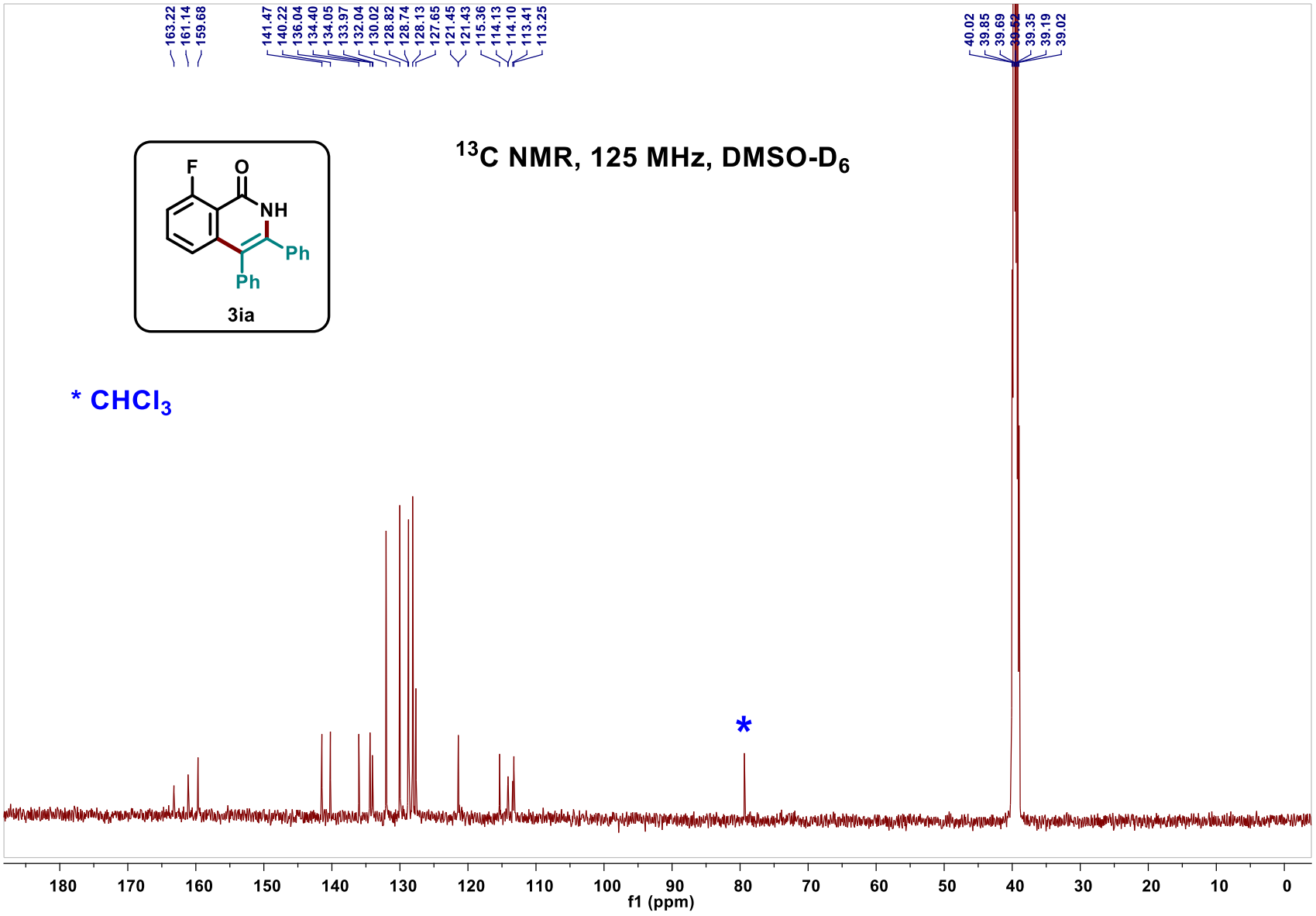
8.58
8.58
7.76
7.75
7.74
7.73
7.57
7.55
7.26
7.26
2.76
2.75
2.74
2.74
2.73
2.72
2.71
2.71
1.36
1.35
1.33
-0.01
1H NMR, 500 MHz, CDCl3
Br
O
NH
Et
Et
3gb
1.02
0.92
1.00
1.00
4.09
3.14
3.09
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

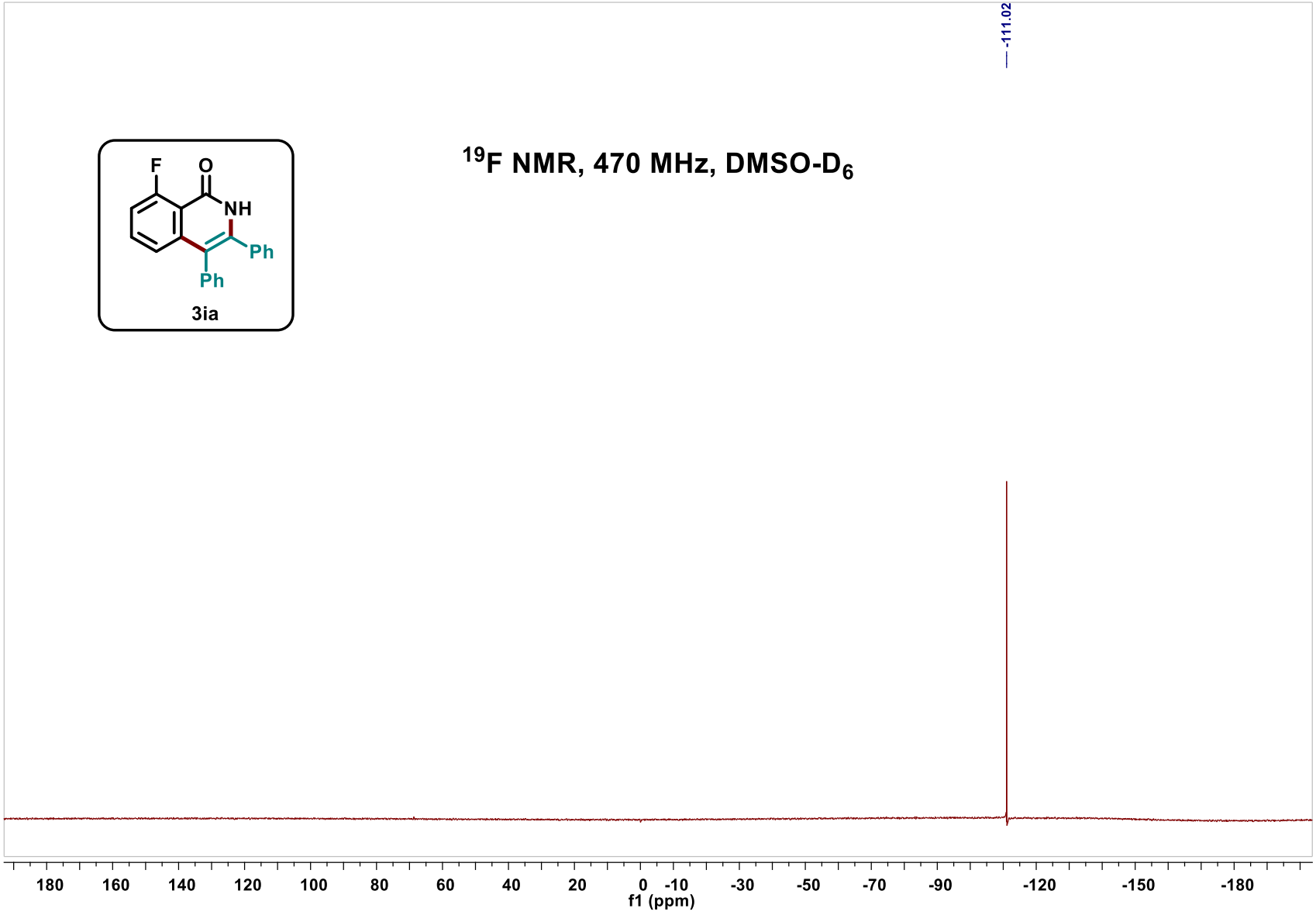

162.87
139.99
136.98
135.43
130.19
126.49
124.78
119.12
113.67
77.25
77.00
76.75
24.24
19.49
14.89
14.01
13C NMR, 125 MHz, CDCl3
Br
O
NH
Et
Et
3gb
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


11.30
8.42
8.41
7.65
7.63
7.62
7.62
7.60
7.60
7.26
2.76
2.75
2.73
2.72
2.70
1.36
1.34
1.33
1.21
1.20
1.18
O
Cl
NH
Et
Et
3hb
1H NMR, 500 MHz, CDCl3
*Other regioisomer
8.49
8.49
8.47
8.47
8.42
8.41
7.72
7.72
7.71
7.71
7.65
7.63
7.62
7.62
7.60
7.60
7.33
7.32
7.30
7.26
0.06
0.93
0.06
2.00
0.06
f1 (ppm)
1.01
0.93
2.00
0.06
4.12
3.15
3.01
f1 (ppm)

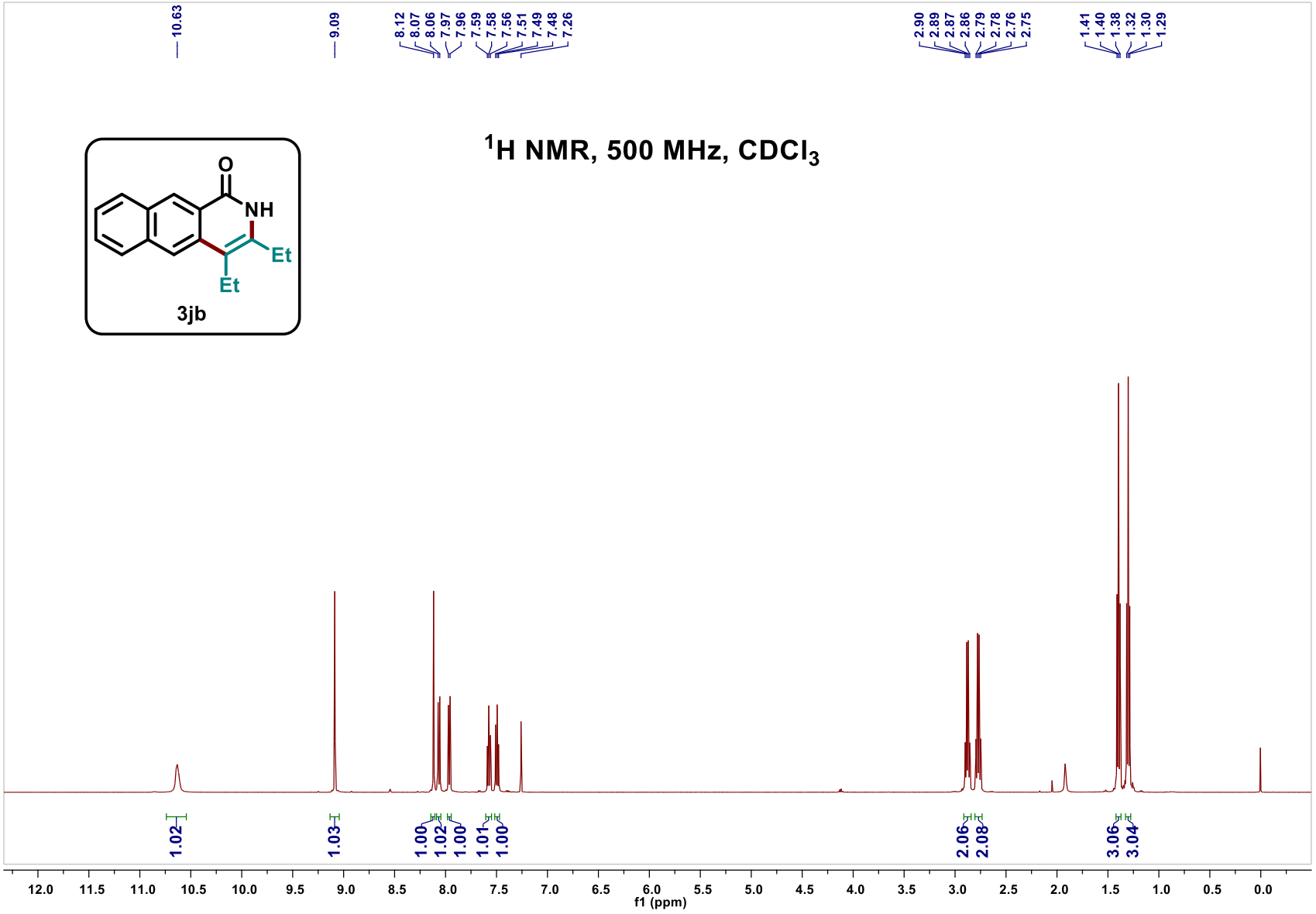

162.74
139.44
136.73
132.81
131.50
127.20
126.47
124.65
113.62
77.25
77.00
76.75
24.25
19.57
14.82
13.79
O
Cl
NH
Et
Et
3hb
^{13}C NMR, 125 MHz, CDCl$_3$
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

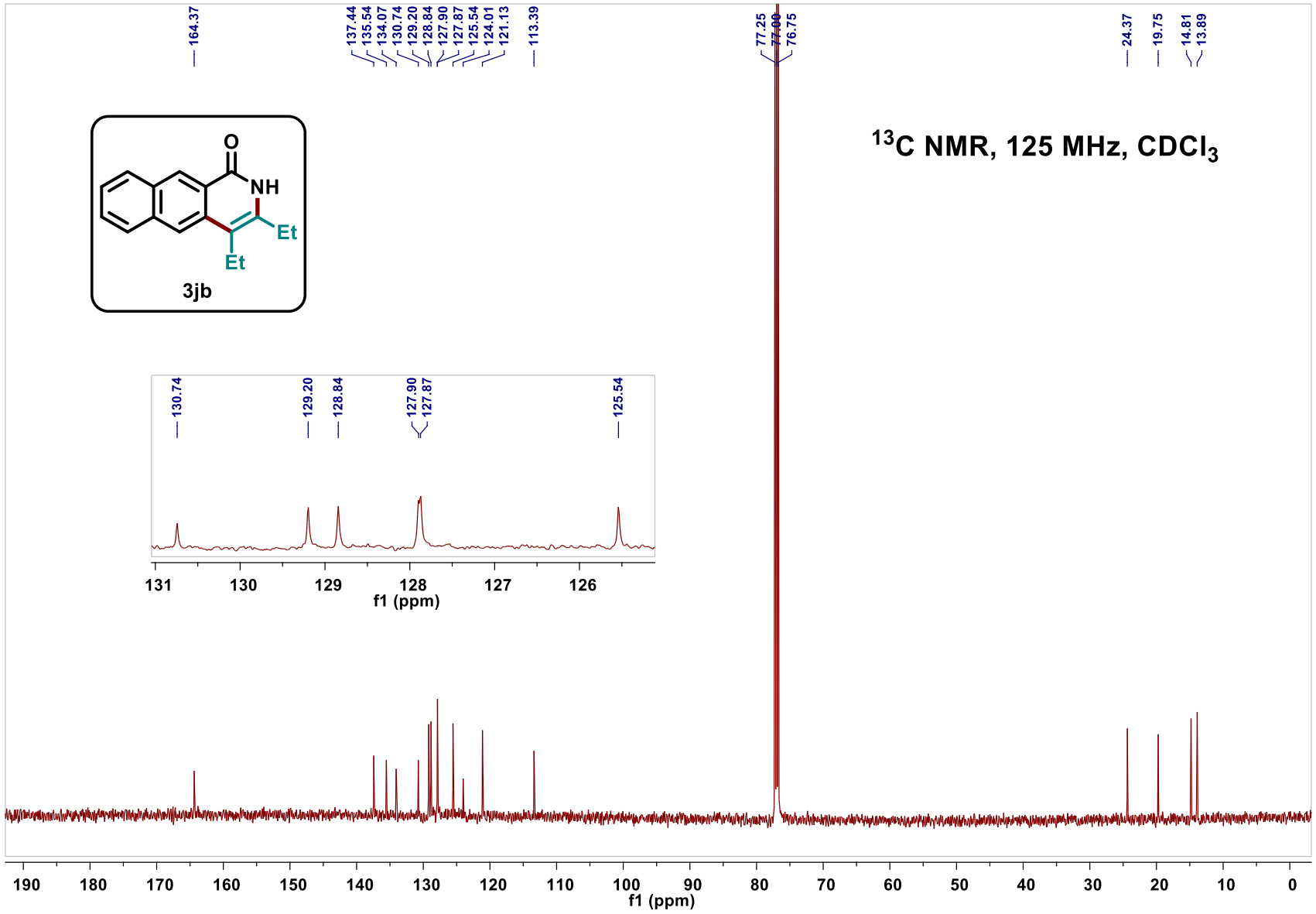

11.56
7.62
7.61
7.60
7.59
7.59
7.58
7.31
7.29
7.28
7.27
7.26
7.25
7.25
7.24
7.23
7.22
7.21
7.15
7.15
7.13
6.91
6.90
3.35
2.50
1H NMR, 500 MHz, DMSO-D6
F
O
NH
Ph
Ph
3ia
* CHCl3
*
0.98
1.00
9.00
2.01
0.97
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

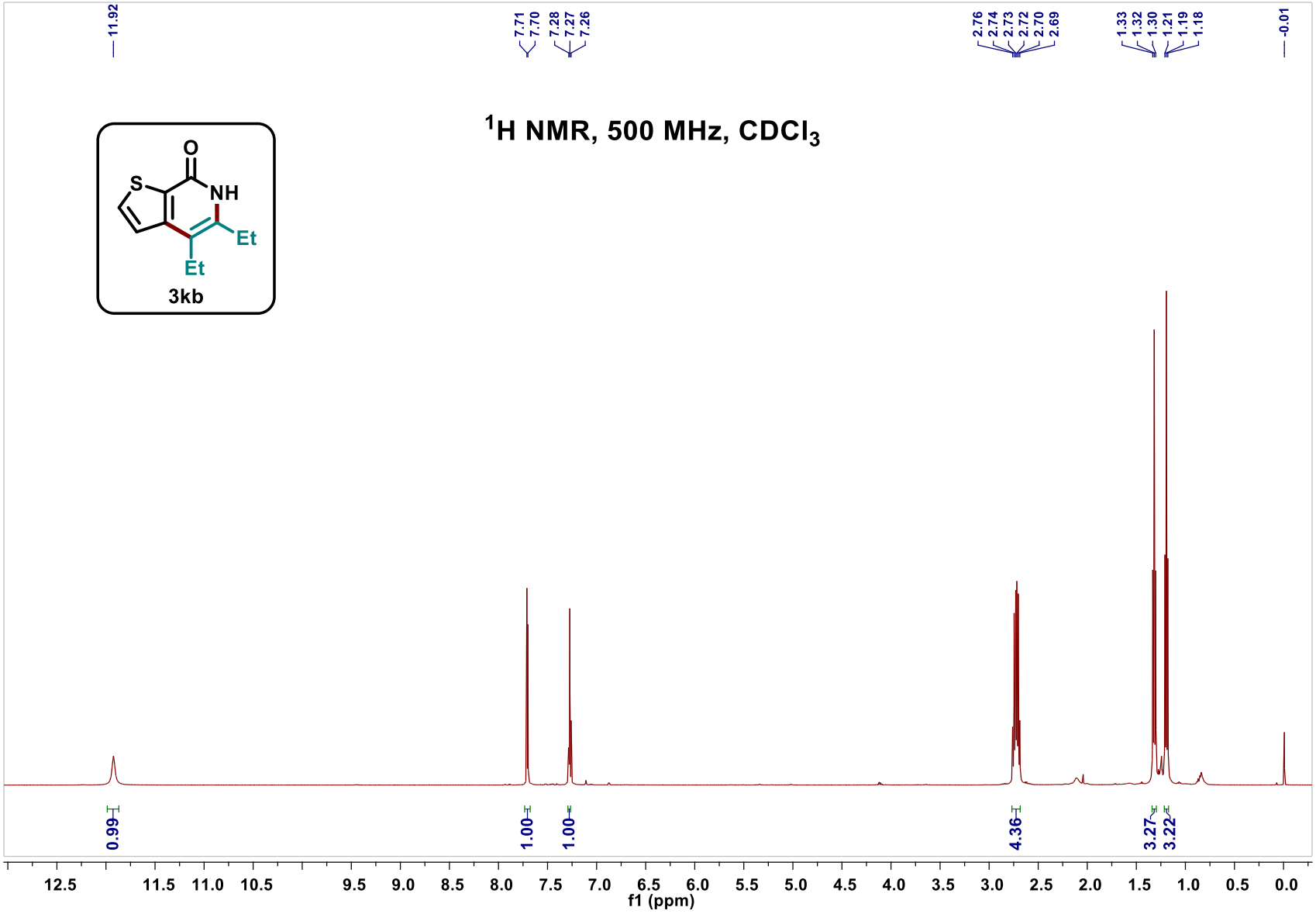

163.22
161.14
159.68
141.47
140.22
136.04
134.40
134.05
133.97
132.04
130.02
128.82
128.74
128.13
127.65
121.45
121.43
115.36
114.13
114.10
113.41
113.25
40.02
39.85
39.69
39.52
39.35
39.19
39.02
F
O
NH
Ph
Ph
3ia
13C NMR, 125 MHz, DMSO-D6
* CHCl3
*
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

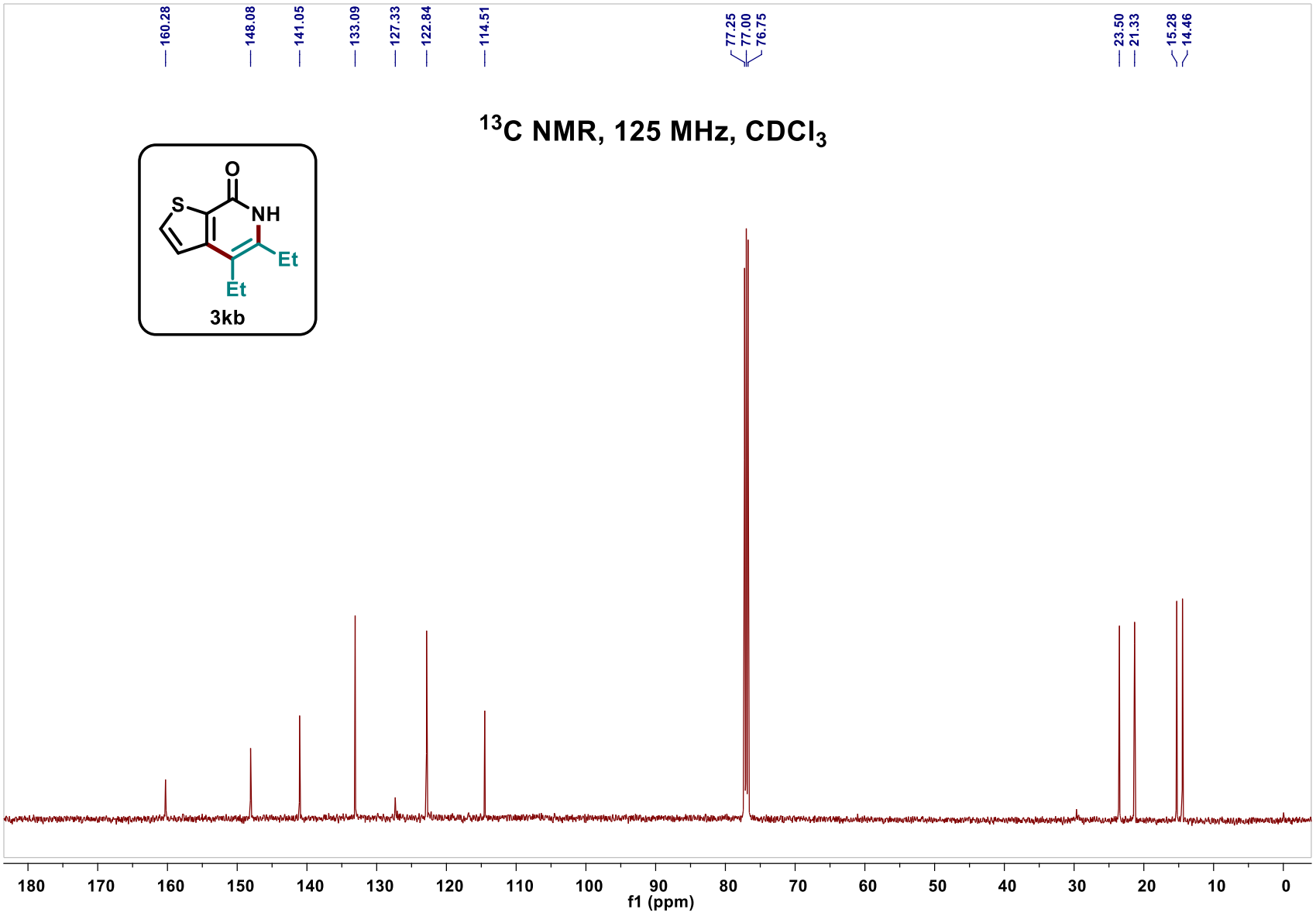


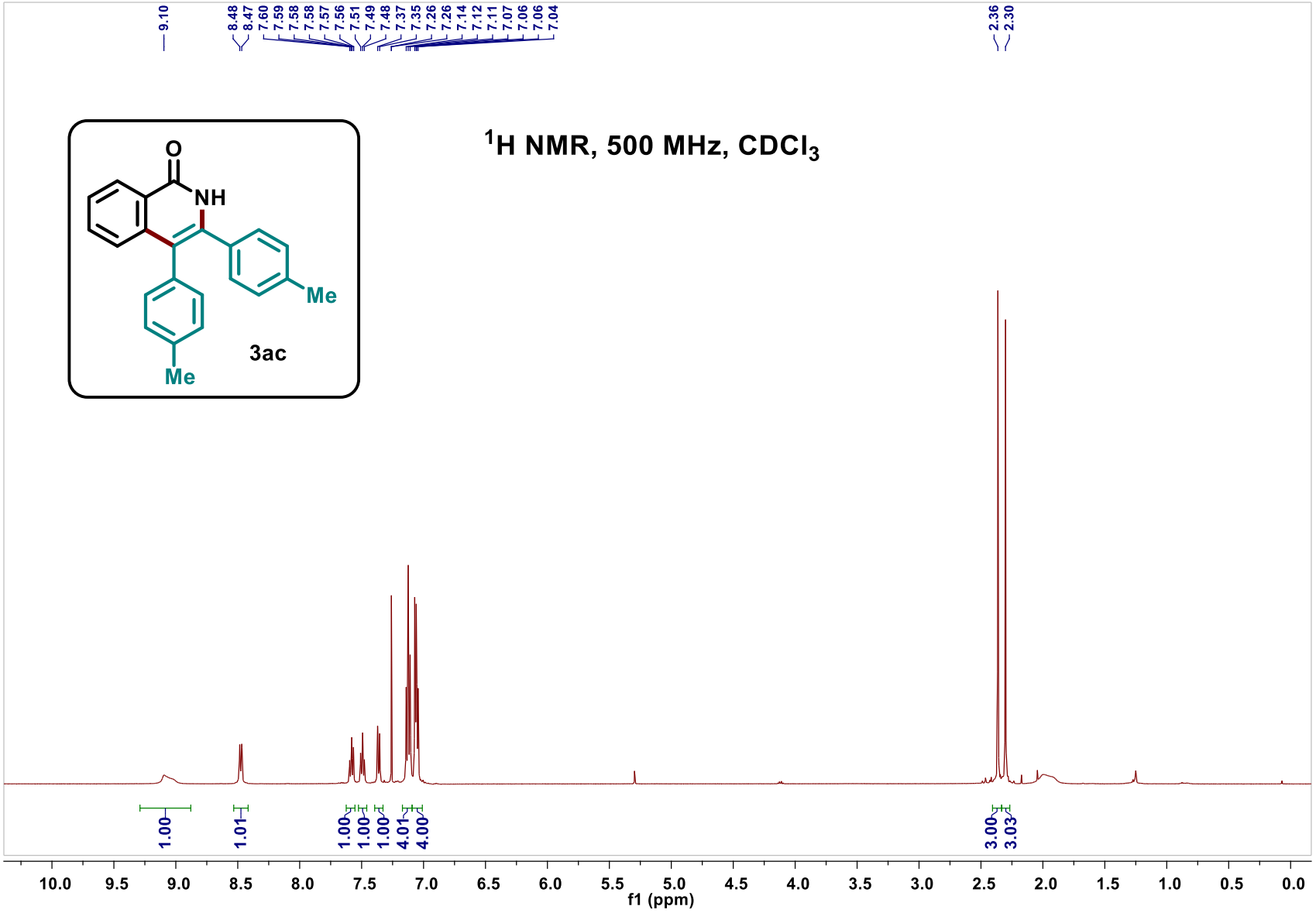
^{19}F NMR, 470 MHz, DMSO-D$_6$

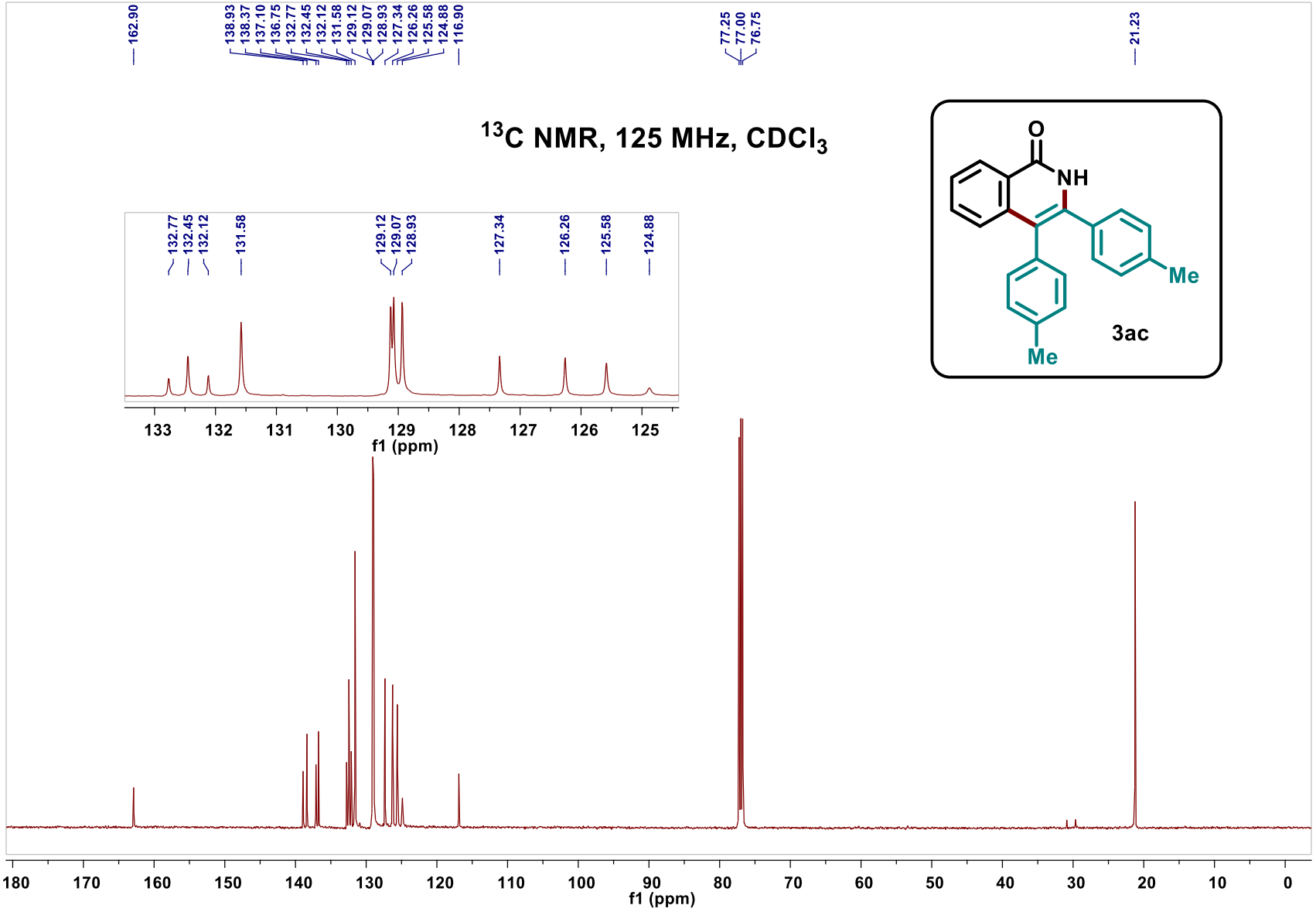

10.63
9.09
8.12
8.07
8.06
7.97
7.96
7.59
7.58
7.56
7.51
7.49
7.48
7.26
2.90
2.89
2.87
2.86
2.79
2.78
2.76
2.75
1.41
1.40
1.38
1.32
1.30
1.29
^{1}H NMR, 500 MHz, CDCl$_3$
O
NH
Et
Et
3jb
1.02
1.03
1.00
1.02
1.00
1.01
1.00
2.06
2.08
3.06
3.04
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

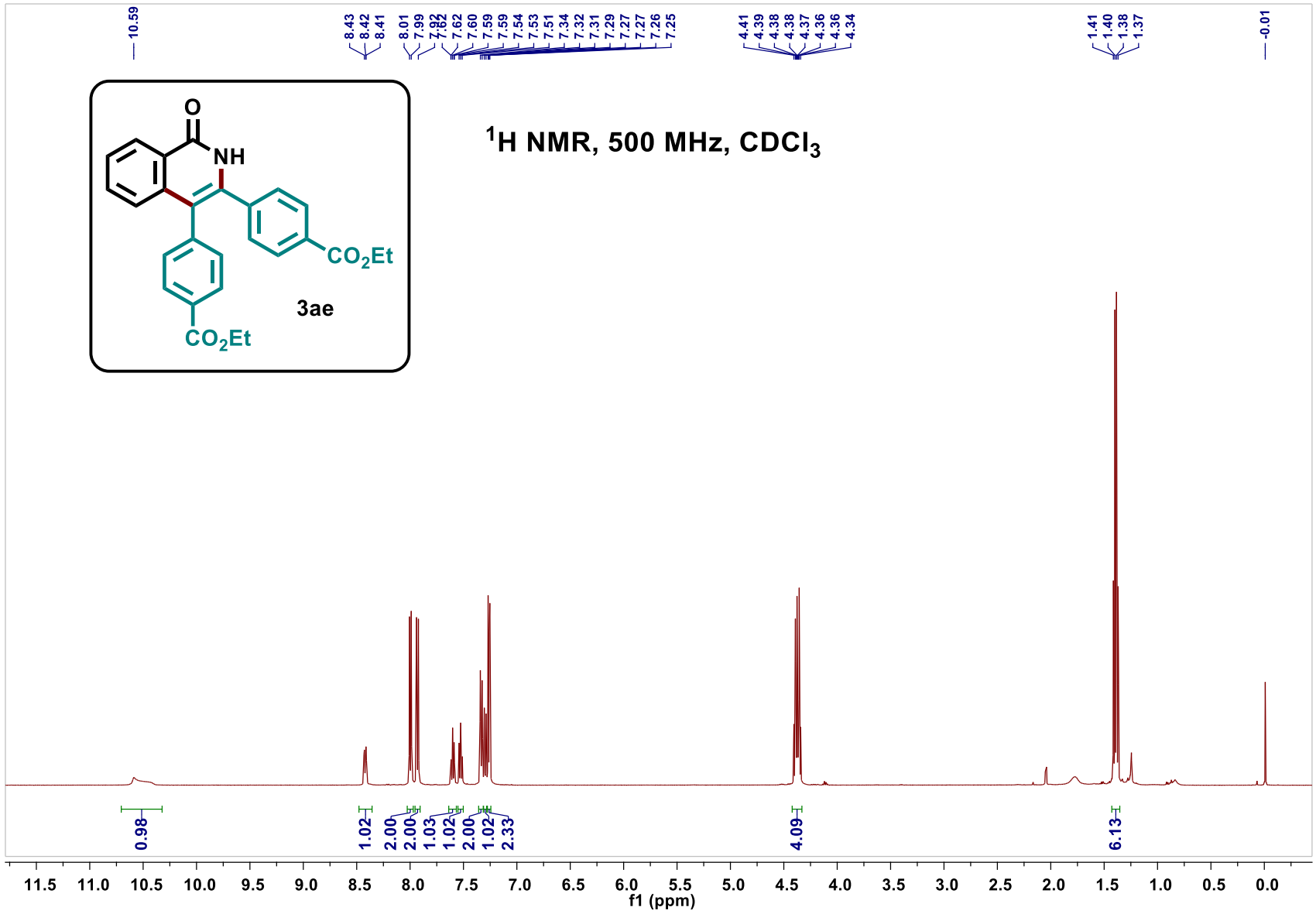

164.37
137.44
135.54
134.07
130.74
129.20
128.84
127.90
127.87
125.54
124.01
121.13
113.39
77.25
77.00
76.75
24.37
19.75
14.81
13.89
O
NH
Et
Et
3jb
^{13}C NMR, 125 MHz, CDCl$_3$
130.74
129.20
128.84
127.90
127.87
125.54
131
130
129
128
127
126
f1 (ppm)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

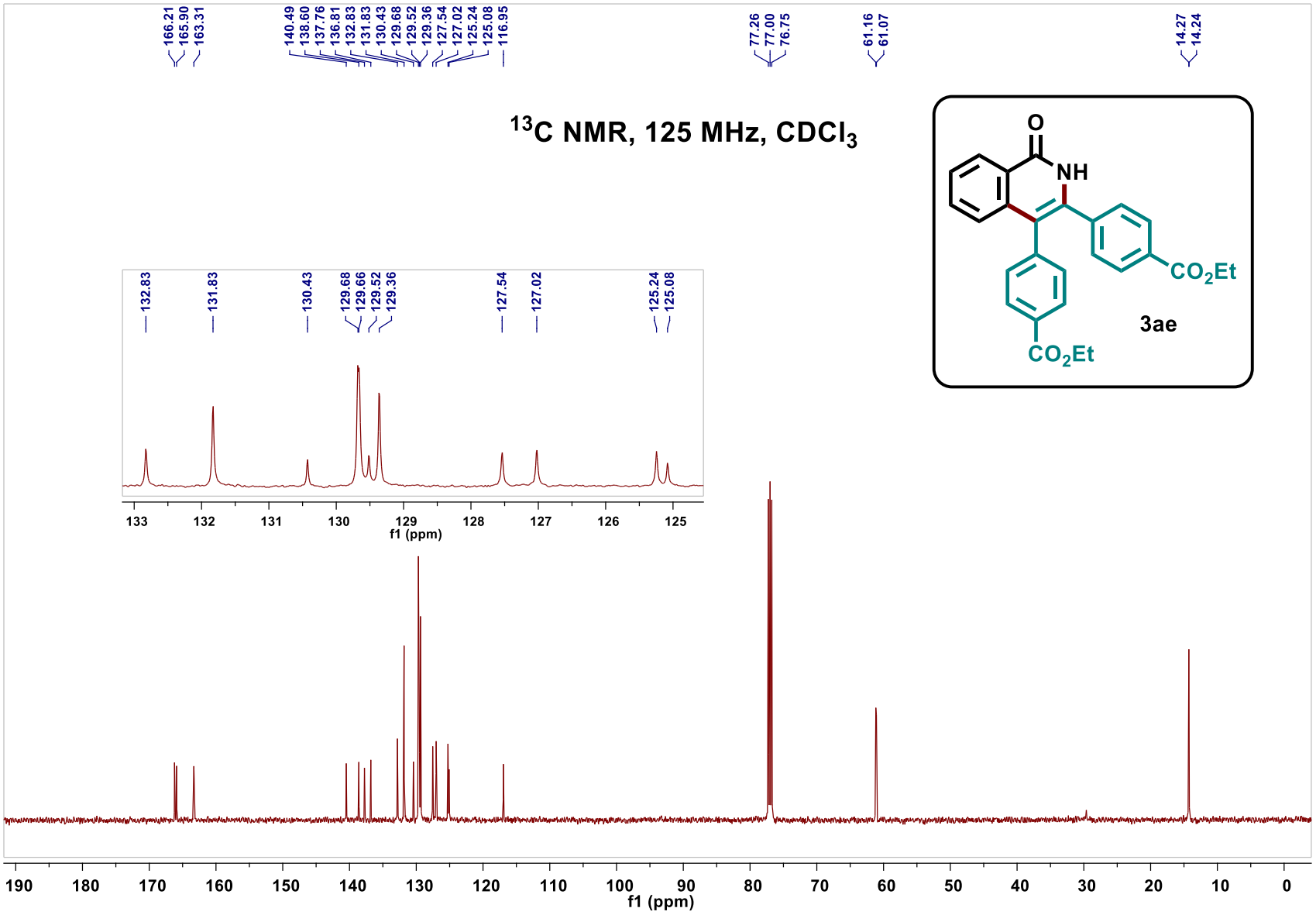

11.92
7.71
7.70
7.28
7.27
7.26
2.76
2.74
2.73
2.72
2.70
2.69
1.33
1.32
1.30
1.21
1.19
1.18
-0.01
1H NMR, 500 MHz, CDCl3
O
S
NH
Et
Et
3kb
0.99
1.00
1.00
4.36
3.27
3.22
12.5
11.5
11.0
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

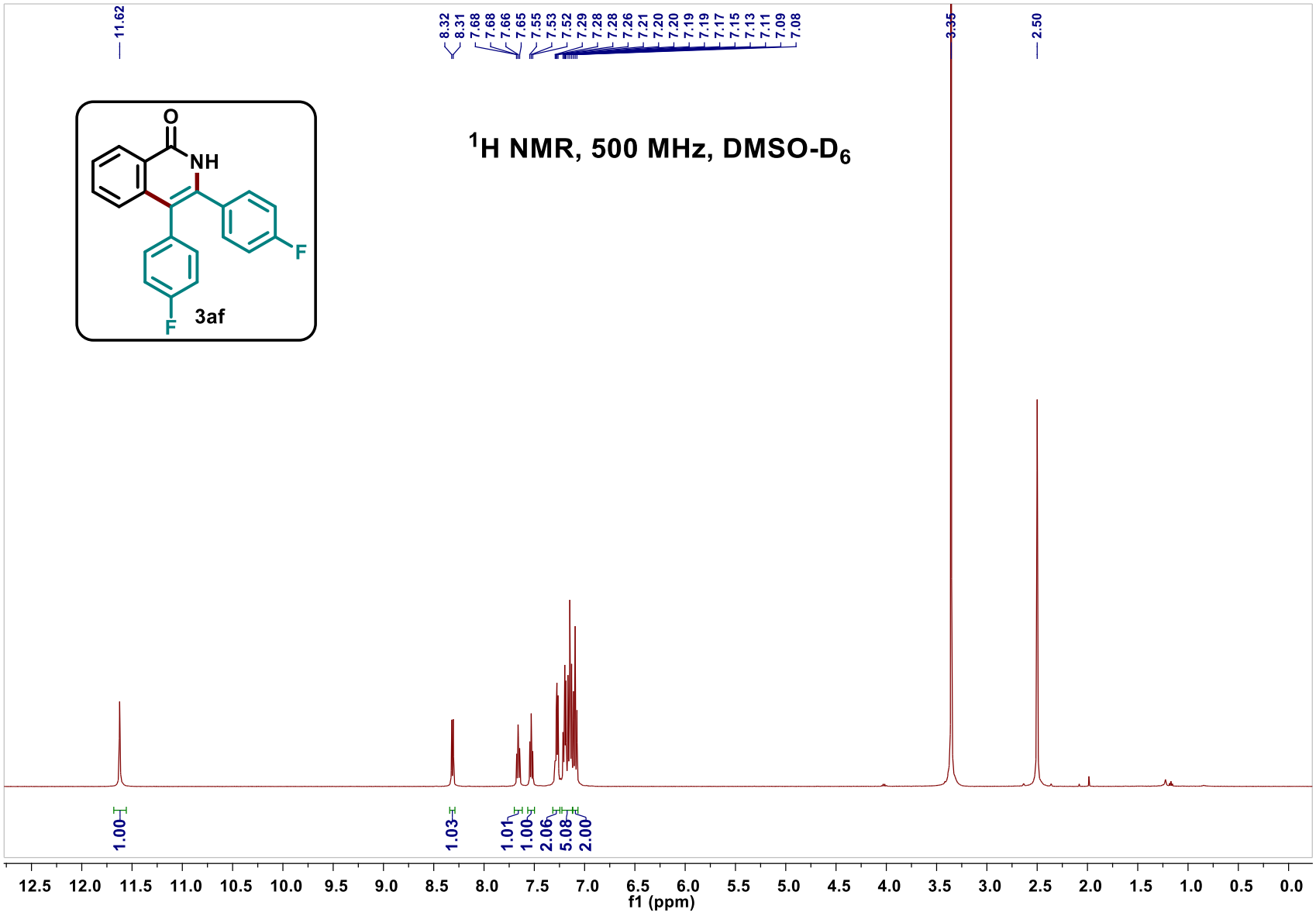

160.28
148.08
141.05
133.09
127.33
122.84
114.51
77.25
77.00
76.75
23.50
21.33
15.28
14.46
13C NMR, 125 MHz, CDCl3
O
S
NH
Et
Et
3kb
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

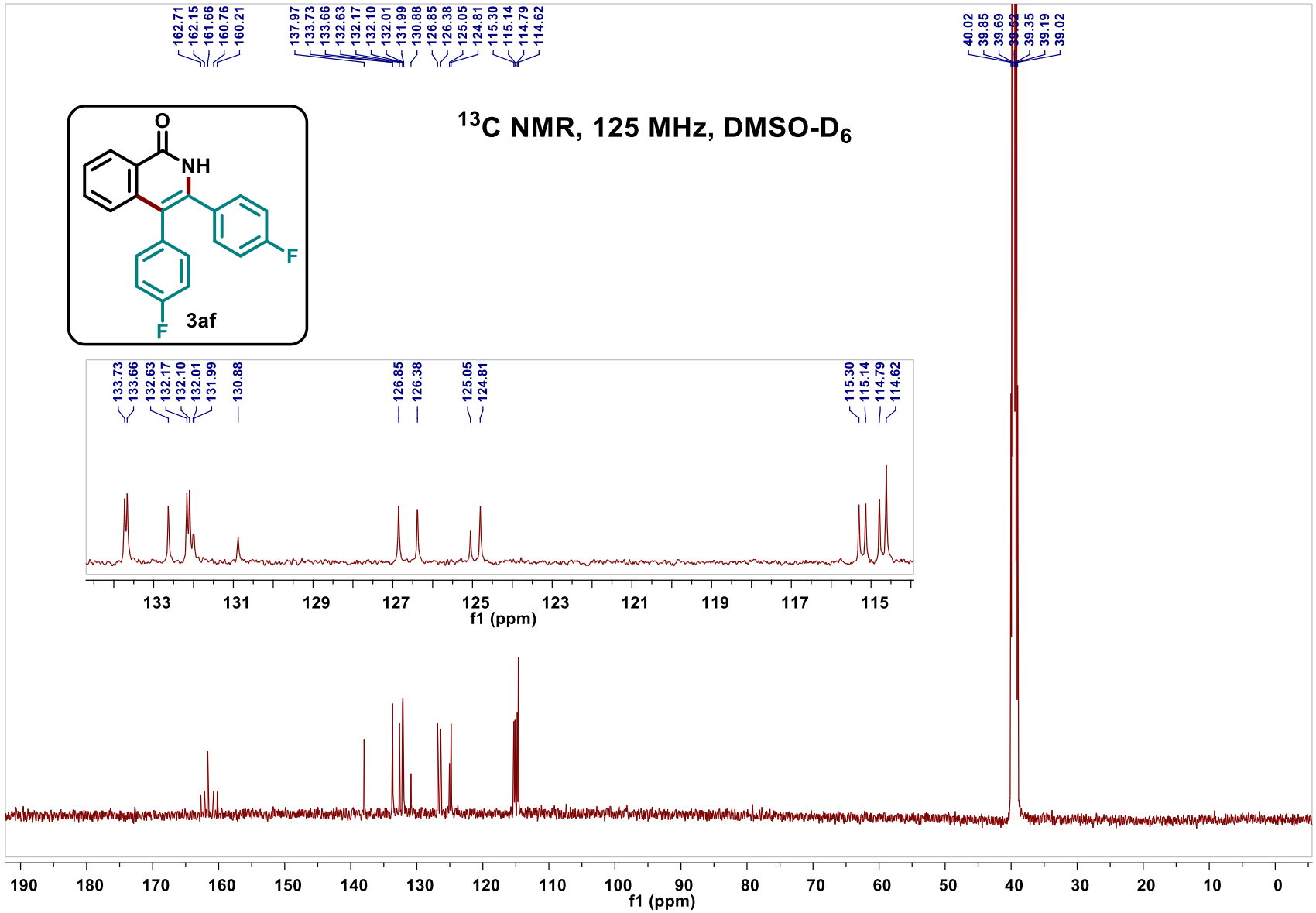

9.10
8.48
8.47
7.60
7.59
7.58
7.58
7.57
7.56
7.51
7.49
7.48
7.37
7.35
7.26
7.26
7.14
7.12
7.11
7.07
7.06
7.06
7.04
2.36
2.30
O
NH
Me
Me
3ac
1H NMR, 500 MHz, CDCl3
1.00
1.01
1.00
1.00
1.00
4.01
4.00
3.00
3.03
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

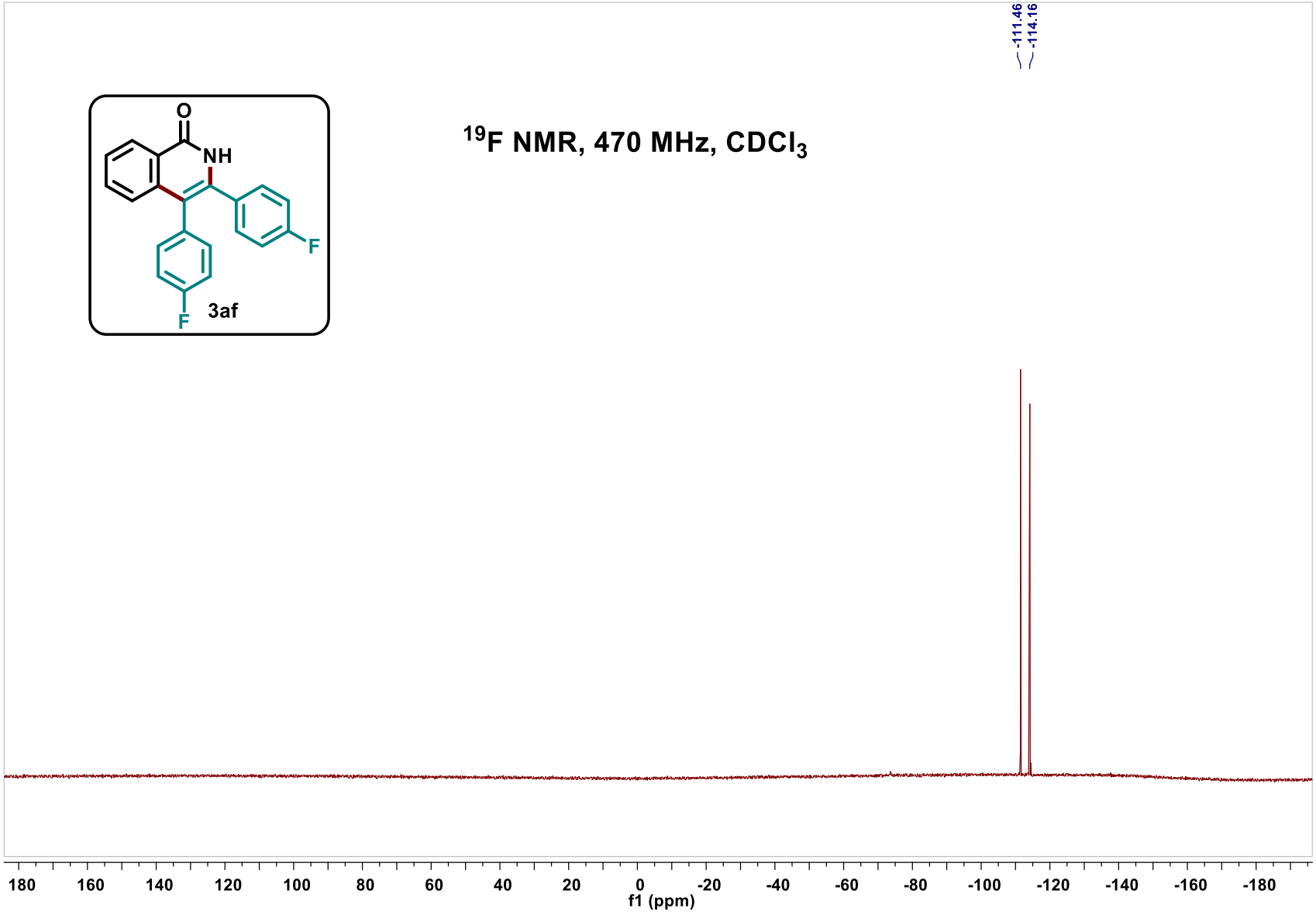

162.90
138.93
138.37
137.10
136.75
132.77
132.45
132.12
131.58
129.12
129.07
128.93
127.34
126.26
125.58
124.88
116.90
77.25
77.00
76.75
21.23
13C NMR, 125 MHz, CDCl3
O
NH
Me
Me
3ac
f1 (ppm)
f1 (ppm)

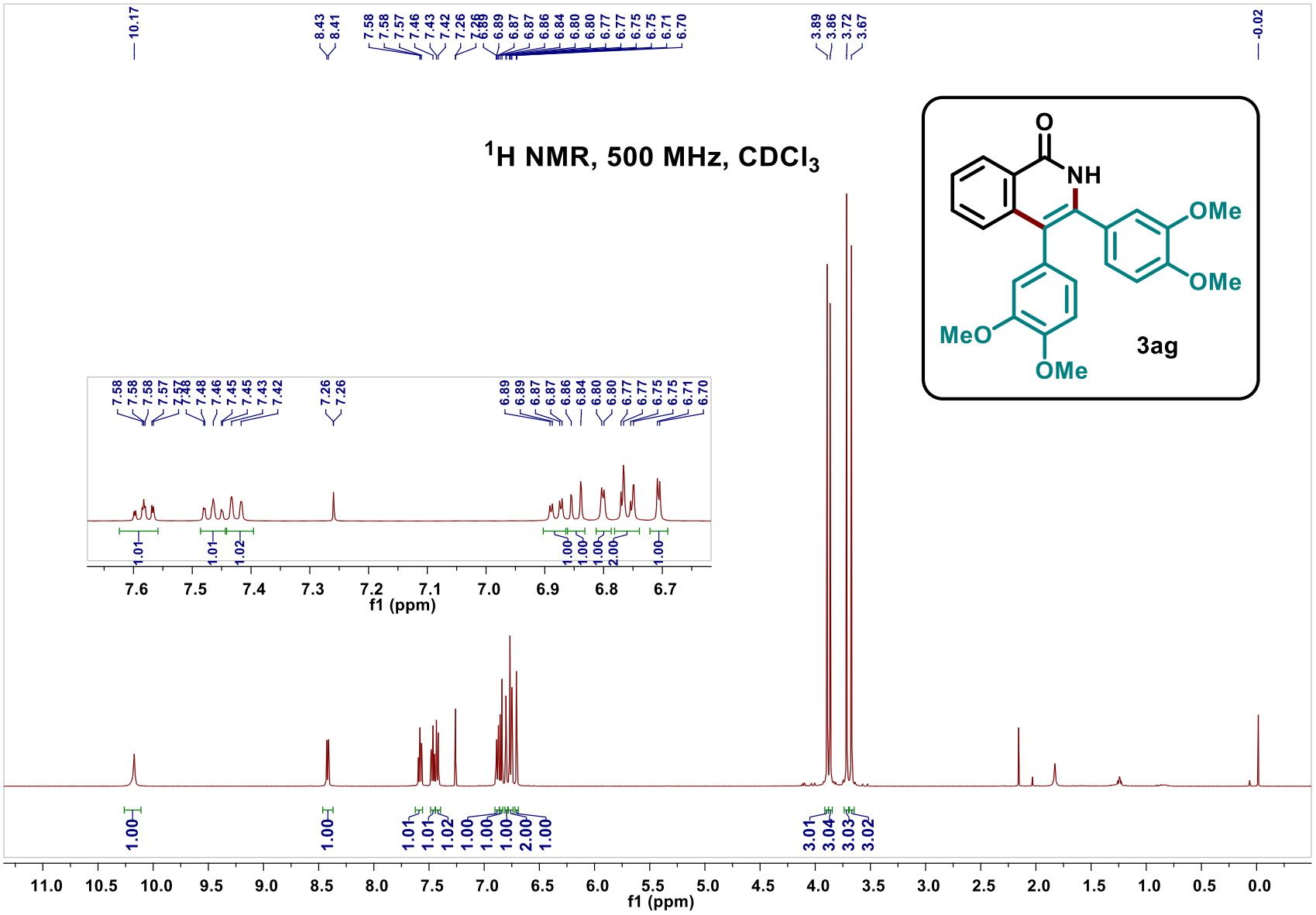

10.59
8.43
8.42
8.41
8.01
7.99
7.82
7.62
7.60
7.59
7.59
7.54
7.53
7.51
7.34
7.32
7.31
7.29
7.27
7.27
7.26
7.25
4.41
4.39
4.38
4.38
4.37
4.36
4.36
4.34
1.41
1.40
1.38
1.37
-0.01
O
NH
CO2Et
CO2Et
3ae
1H NMR, 500 MHz, CDCl3
0.98
1.02
2.00
2.00
1.03
1.02
2.00
1.02
2.33
4.09
6.13
11.5 11.0 10.5 10.0 9.5 9.0 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 0.0
f1 (ppm)

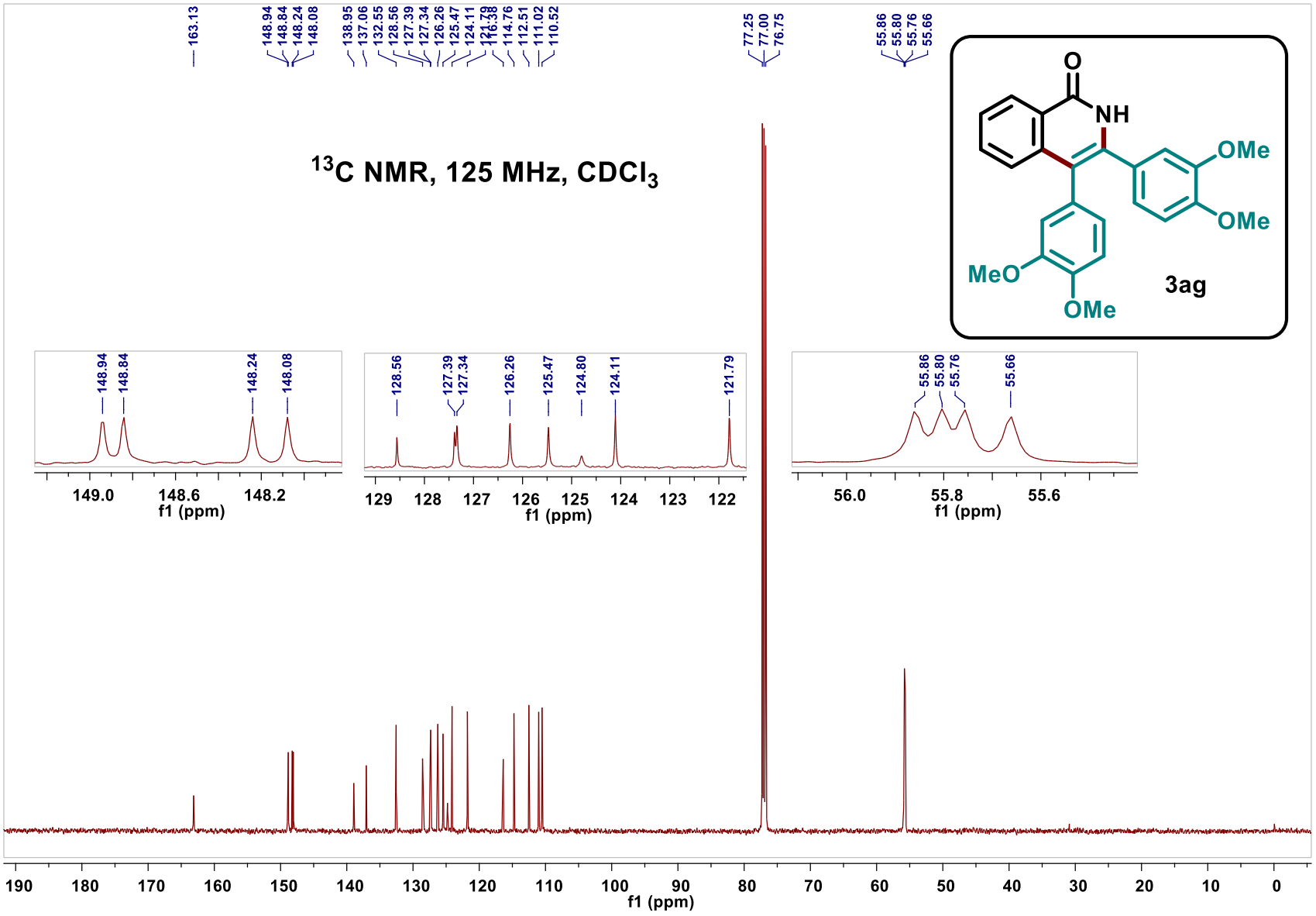

166.21
165.90
163.31
140.49
138.60
137.76
136.81
132.83
131.83
130.43
129.68
129.52
129.36
127.54
127.02
125.24
125.08
116.95
77.26
77.00
76.75
61.16
61.07
14.27
14.24
^{13}C NMR, 125 MHz, CDCl$_3$
O
NH
CO$_2$Et
CO$_2$Et
3ae
132.83
131.83
130.43
129.68
129.66
129.52
129.36
127.54
127.02
125.24
125.08
133
132
131
130
129
128
127
126
125
f1 (ppm)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

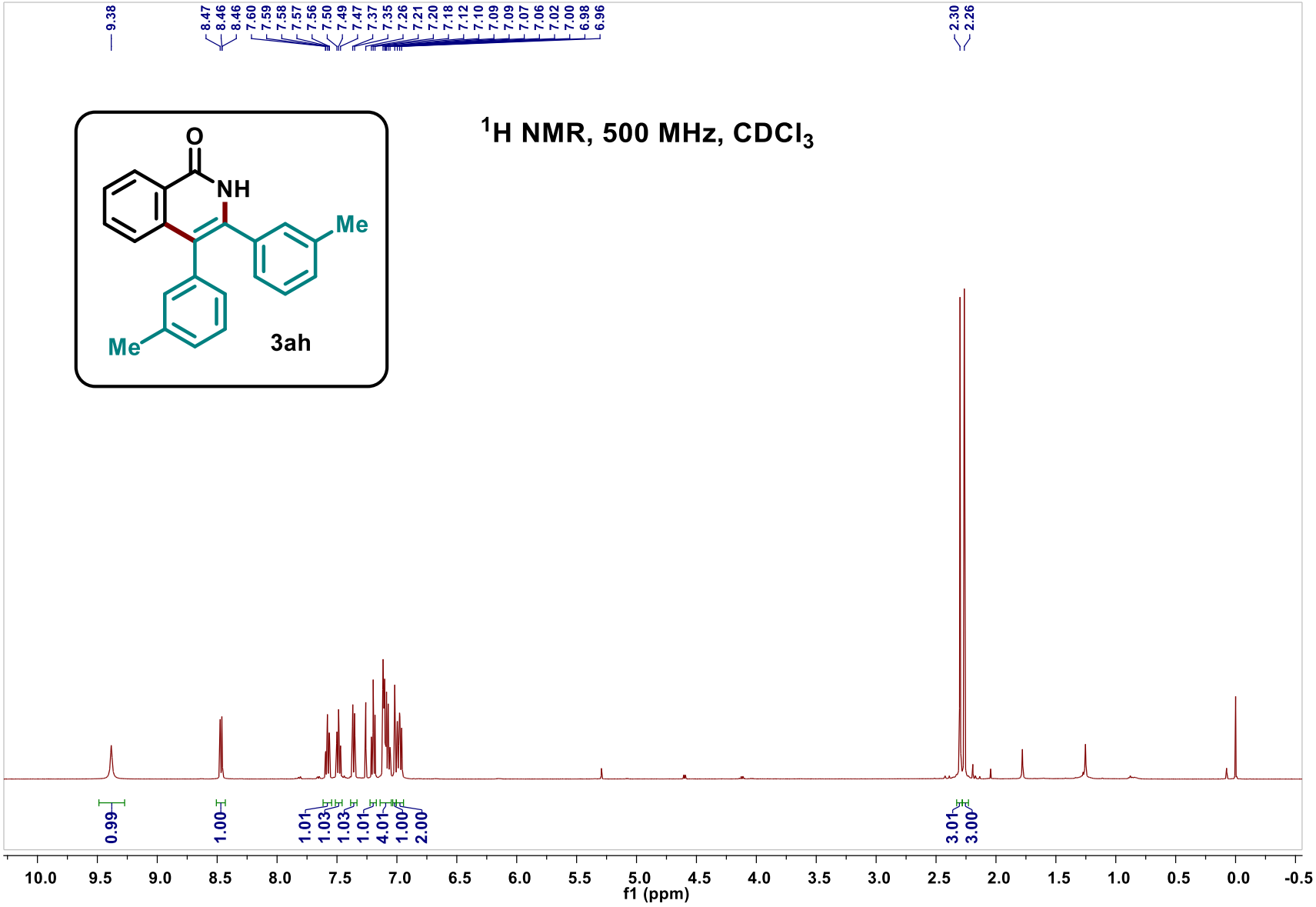

11.62
8.32
8.31
7.68
7.68
7.66
7.65
7.55
7.53
7.52
7.29
7.28
7.28
7.26
7.21
7.20
7.20
7.19
7.19
7.17
7.15
7.13
7.11
7.09
7.08
3.35
2.50
O
NH
F
F
3af
1H NMR, 500 MHz, DMSO-D6
1.00
1.03
1.01
1.00
2.06
5.08
2.00
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

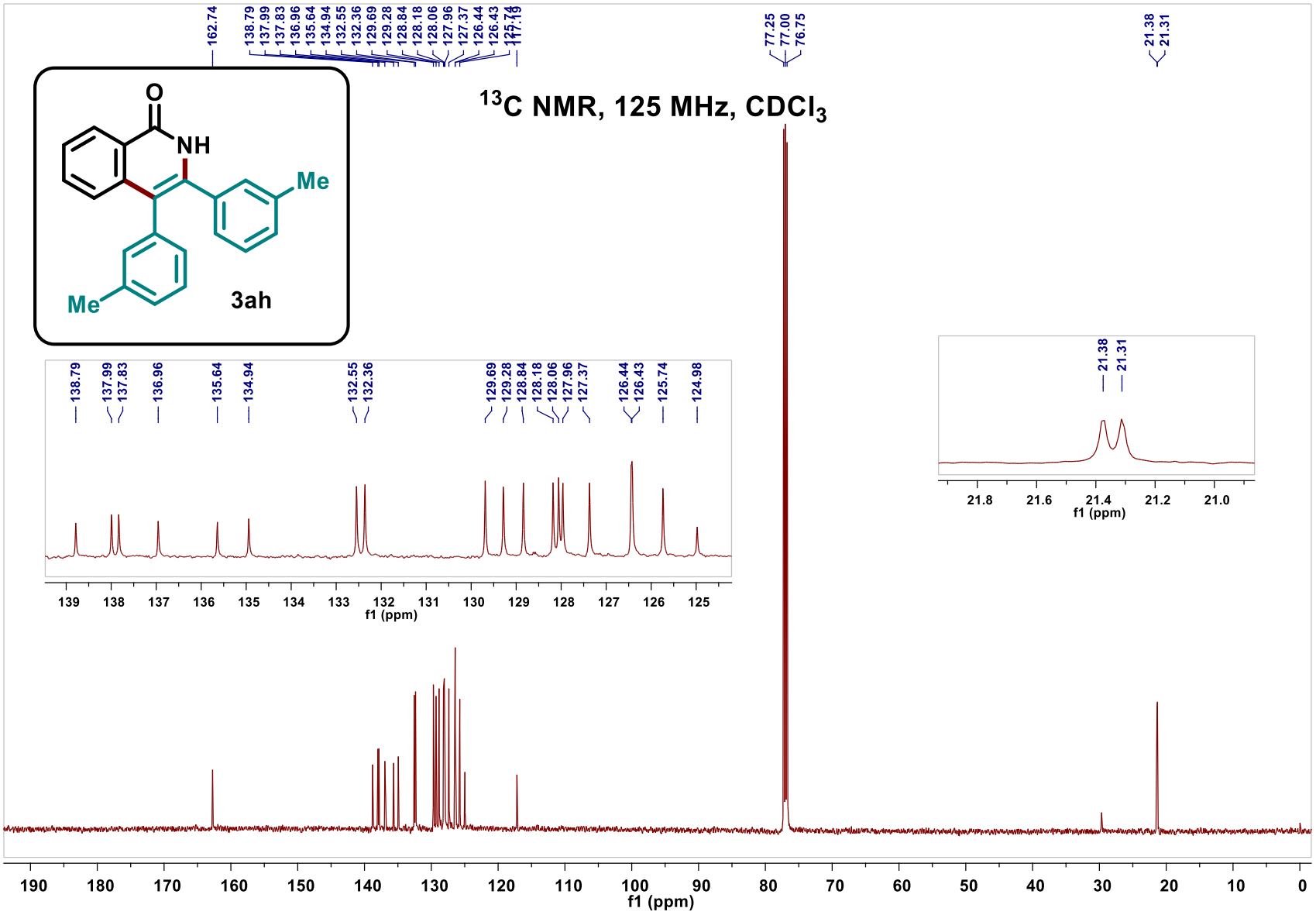

^{13}C NMR, 125 MHz, DMSO-D$_6$
3af
f1 (ppm)
f1 (ppm)

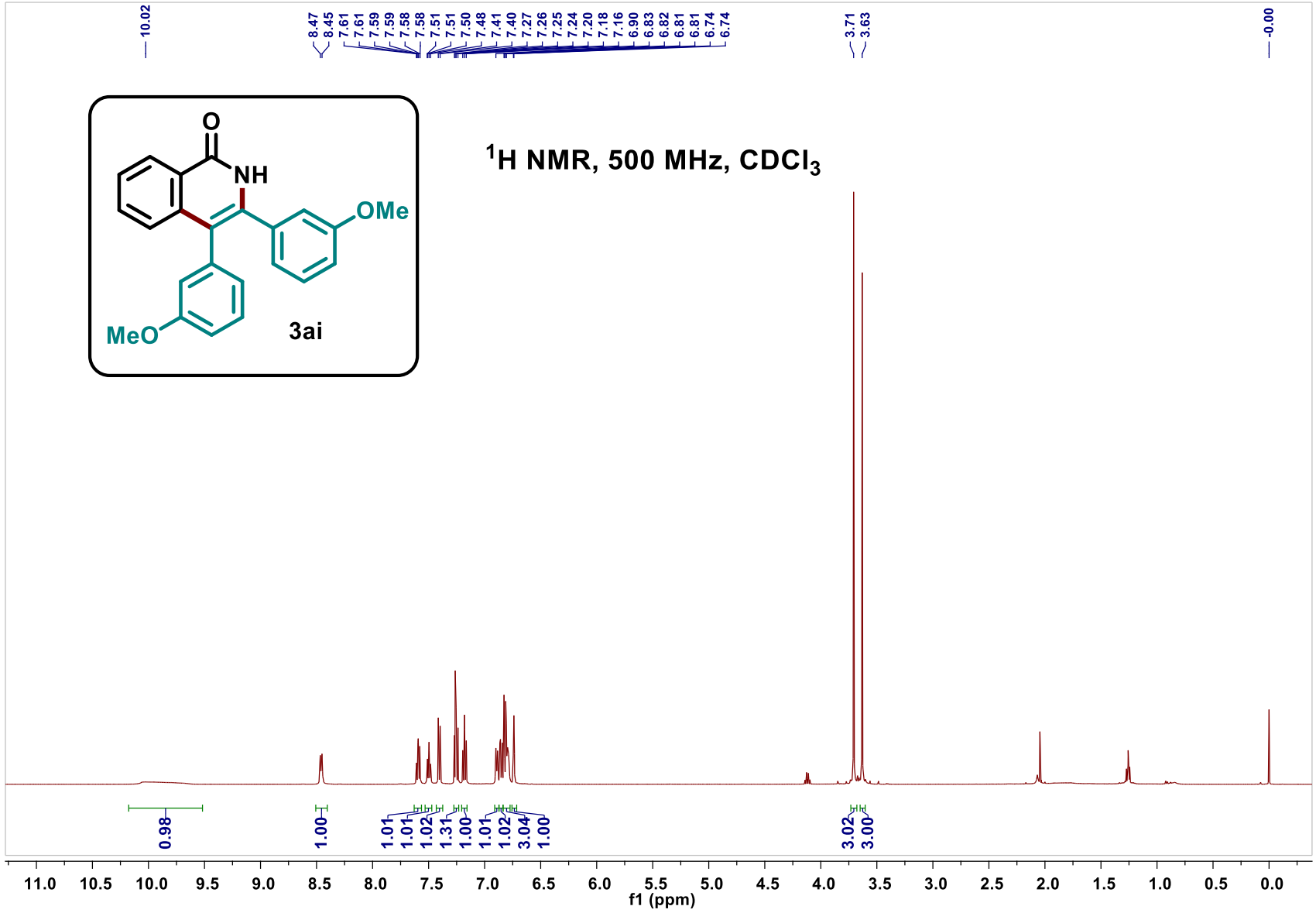

-111.46
-114.16
O
NH
F
F
3af
19F NMR, 470 MHz, CDCl3
180
160
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
f1 (ppm)

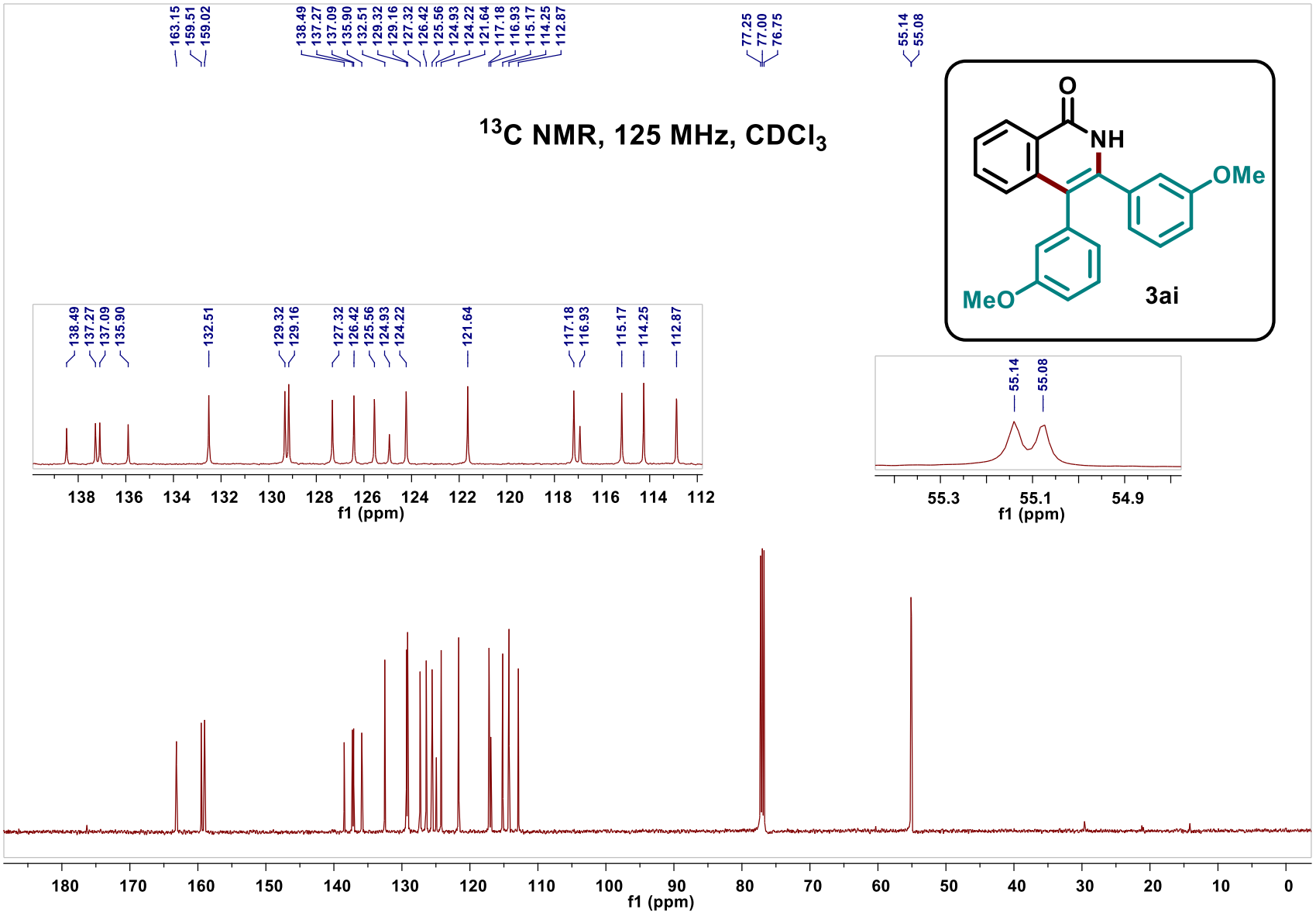

^{1}H NMR, 500 MHz, $CDCl_3$
3ag
NH
OMe
OMe
MeO
OMe
f1 (ppm)

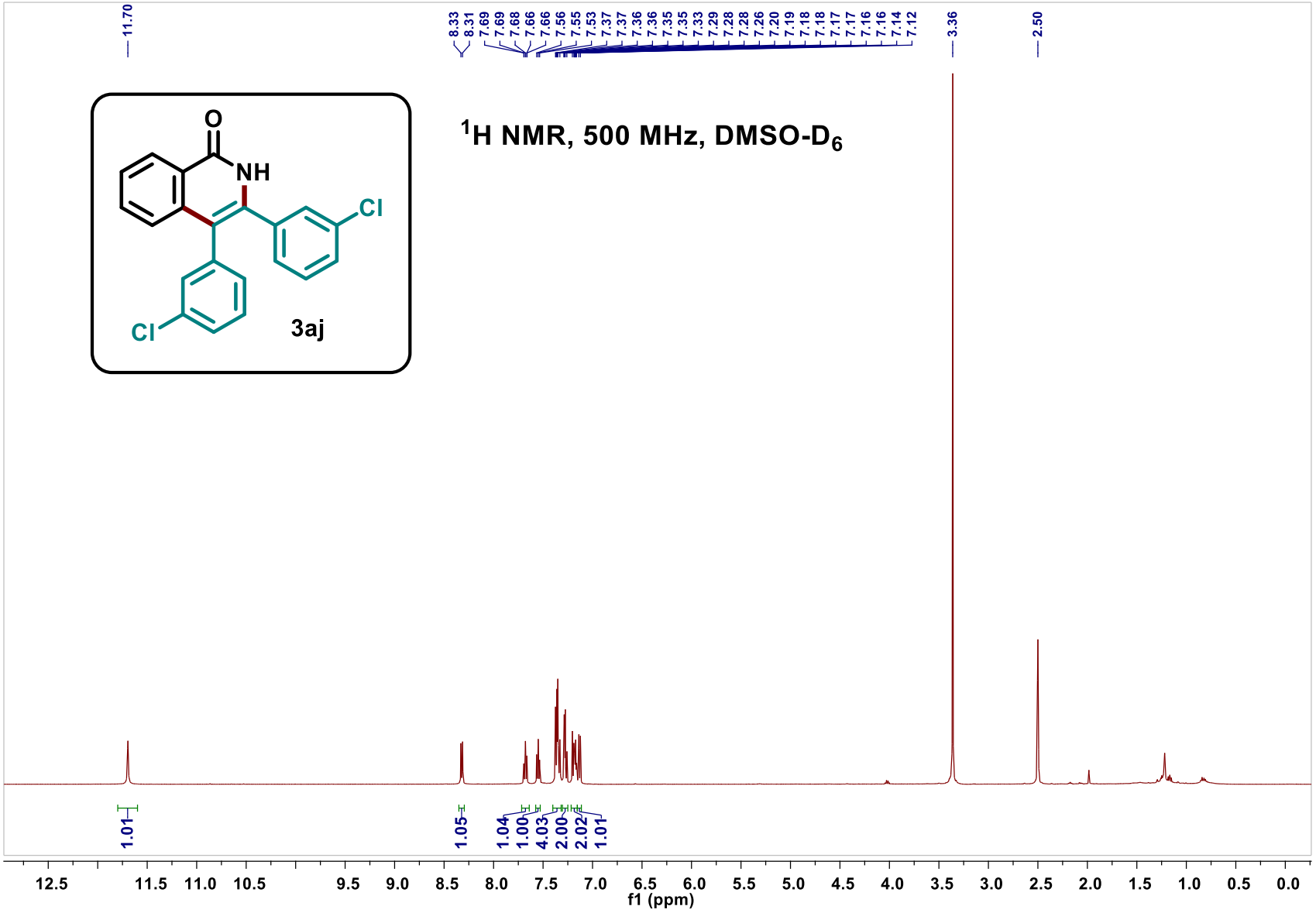

163.13
148.94
148.84
148.24
148.08
138.95
137.06
132.55
128.56
127.39
127.34
126.26
125.47
124.11
121.79
116.38
114.76
112.51
111.02
110.52
77.25
77.00
76.75
55.86
55.80
55.76
55.66
13C NMR, 125 MHz, CDCl3
O
NH
OMe
OMe
MeO
OMe
3ag
f1 (ppm)

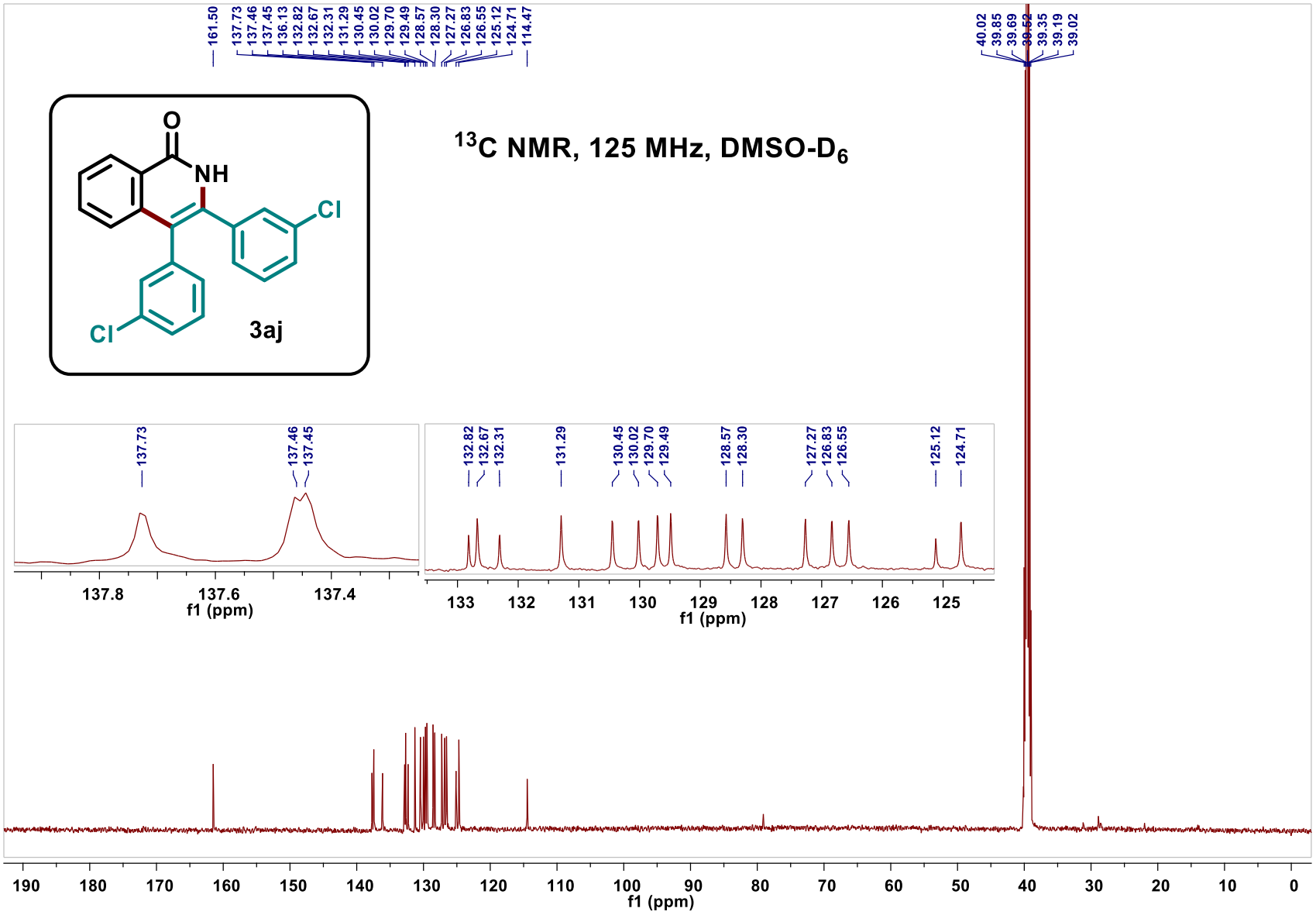

9.38
8.47
8.46
8.46
7.60
7.59
7.58
7.57
7.56
7.50
7.49
7.47
7.37
7.35
7.26
7.21
7.20
7.18
7.12
7.10
7.09
7.09
7.07
7.06
7.02
7.00
6.98
6.96
2.30
2.26
O
NH
Me
Me
3ah
1H NMR, 500 MHz, CDCl3
0.99
1.00
1.01
1.03
1.03
1.01
4.01
1.00
2.00
3.01
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

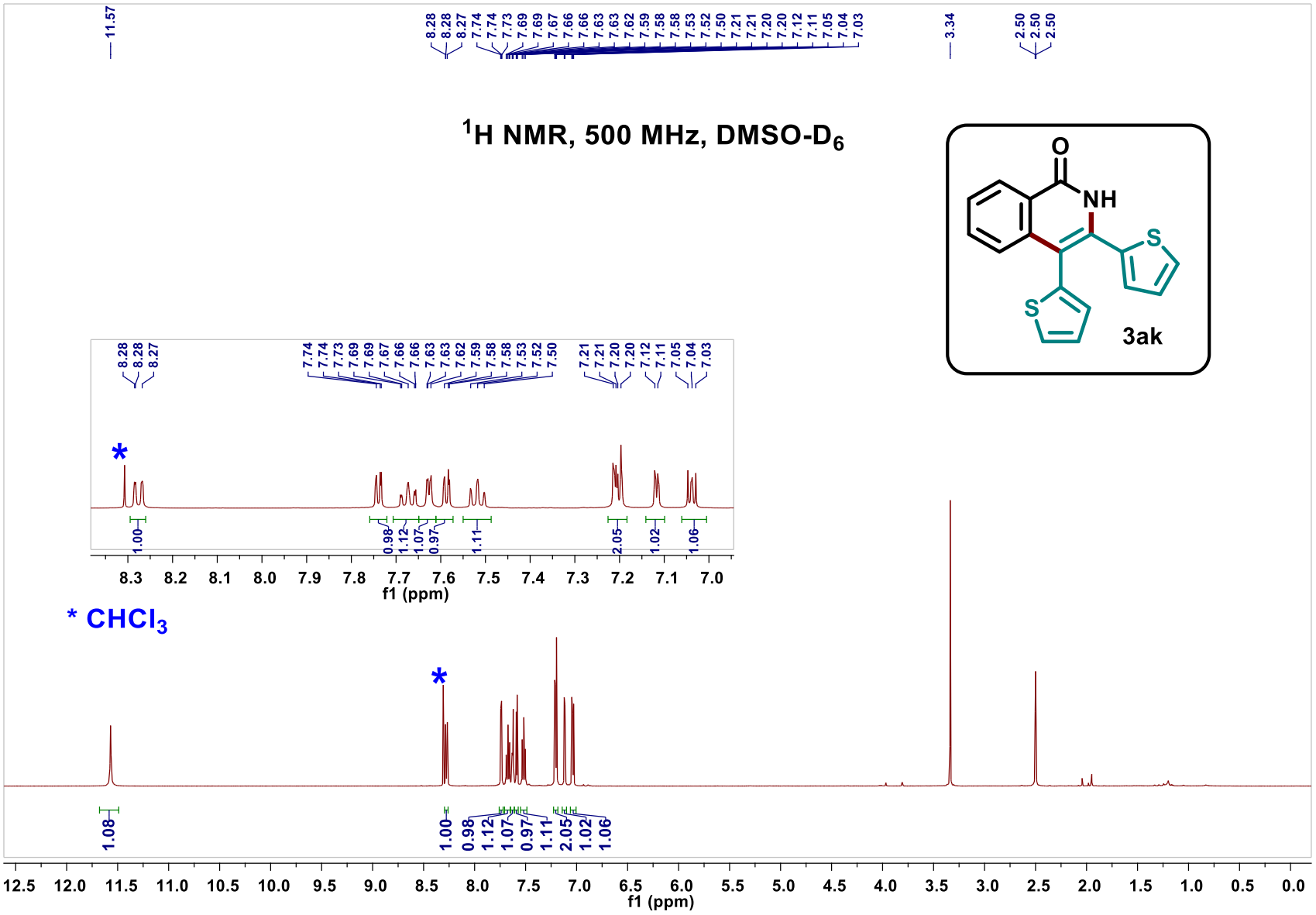

^{13}C NMR, 125 MHz, $CDCl_3$
3ah
Me
Me
NH
O
162.74
138.79
137.99
137.83
136.96
135.64
134.94
132.55
132.36
129.69
129.28
128.84
128.18
128.06
127.96
127.37
126.44
126.43
125.74
124.98
77.25
77.00
76.75
21.38
21.31
f1 (ppm)

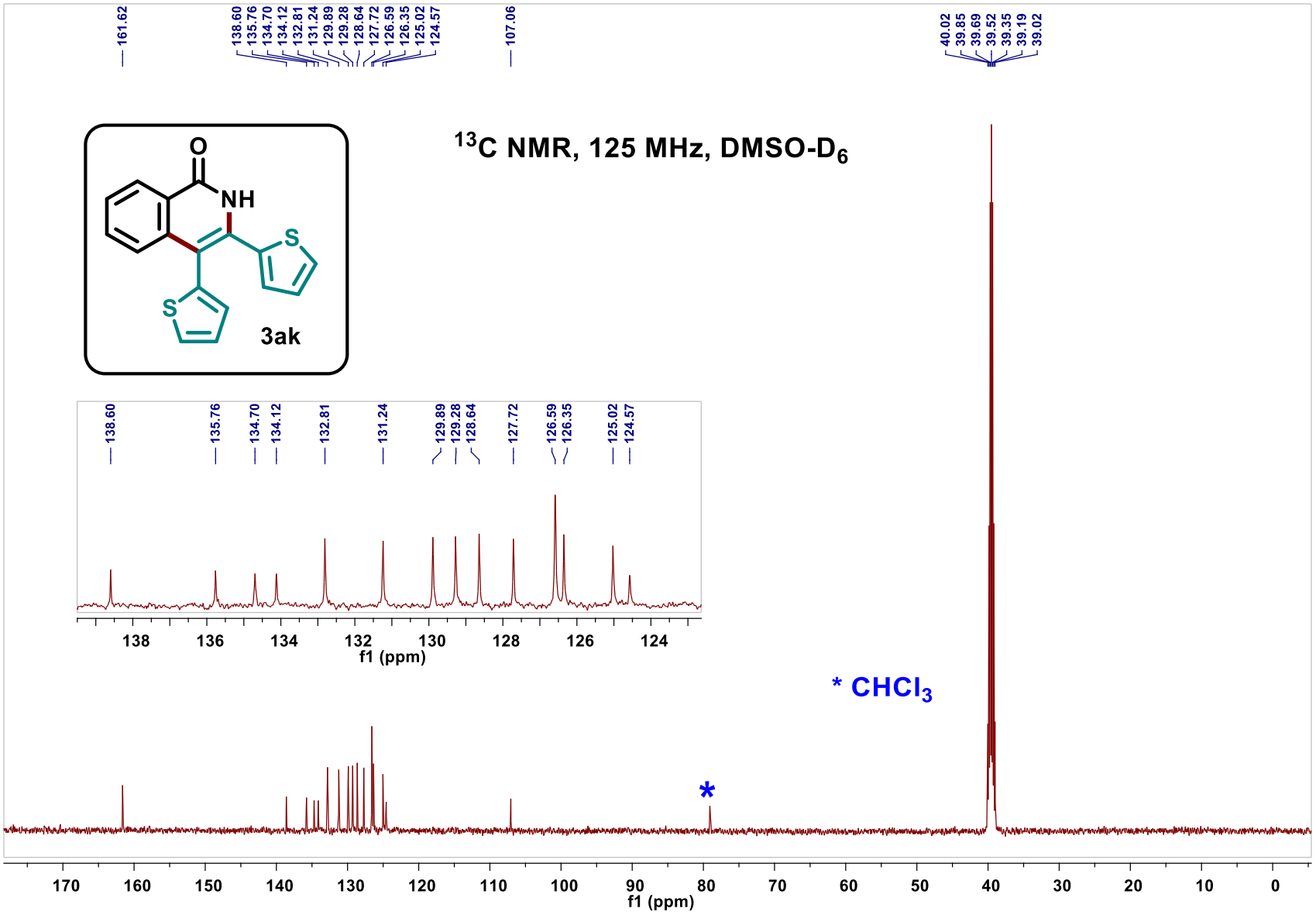

10.02
8.47
8.45
7.61
7.61
7.59
7.59
7.58
7.58
7.51
7.51
7.50
7.48
7.41
7.40
7.27
7.26
7.25
7.24
7.20
7.18
7.16
6.90
6.83
6.82
6.81
6.81
6.74
6.74
3.71
3.63
-0.00
O
NH
OMe
MeO
3ai
1H NMR, 500 MHz, CDCl3
0.98
1.00
1.01
1.01
1.02
1.31
1.00
1.01
1.02
3.04
1.00
3.02
3.00
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
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
0.0
f1 (ppm)

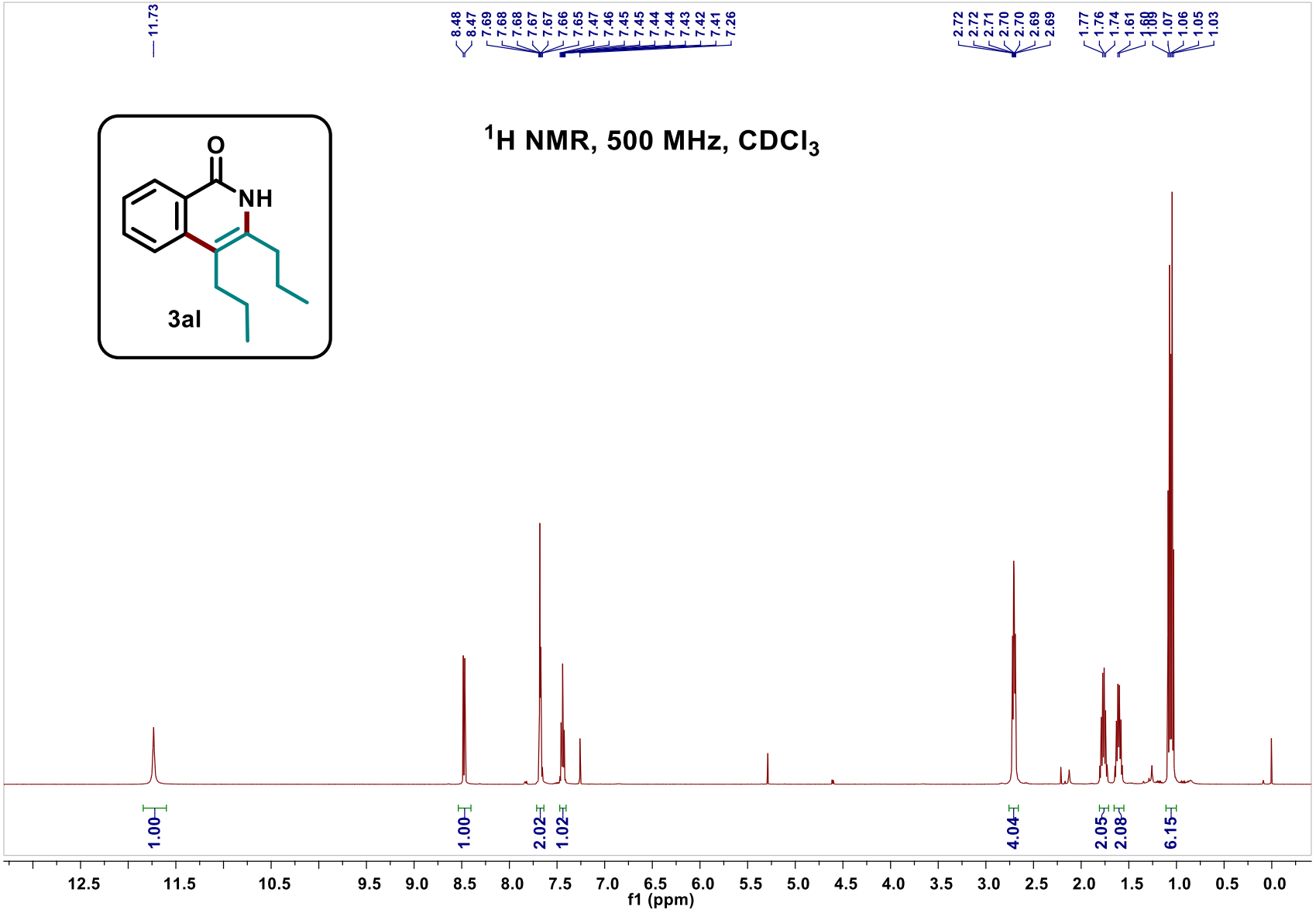

163.15
159.51
159.02
138.49
137.27
137.09
135.90
132.51
129.32
129.16
127.32
126.42
125.56
124.93
124.22
121.64
117.18
116.93
115.17
114.25
112.87
77.25
77.00
76.75
55.14
55.08
13C NMR, 125 MHz, CDCl3
O
NH
OMe
MeO
3ai
f1 (ppm)
f1 (ppm)
f1 (ppm)

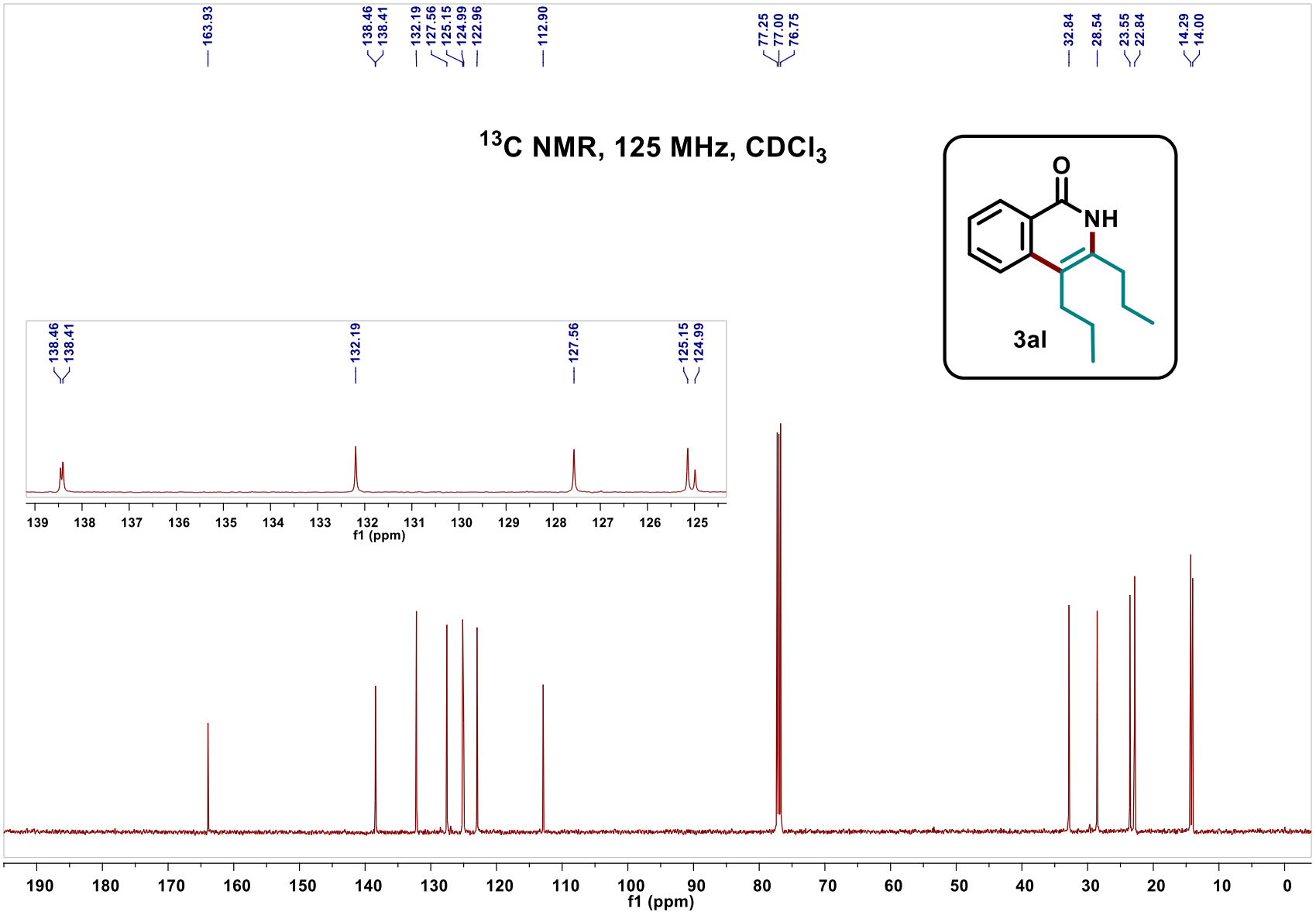

11.70
8.33
8.31
7.69
7.69
7.68
7.66
7.66
7.56
7.55
7.53
7.37
7.37
7.36
7.36
7.35
7.35
7.33
7.29
7.28
7.28
7.26
7.20
7.19
7.18
7.18
7.17
7.17
7.16
7.16
7.14
7.12
3.36
2.50
O
NH
Cl
Cl
3aj
1H NMR, 500 MHz, DMSO-D6
1.01
1.05
1.04
1.00
4.03
2.00
2.02
1.01
12.5
11.5
11.0
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

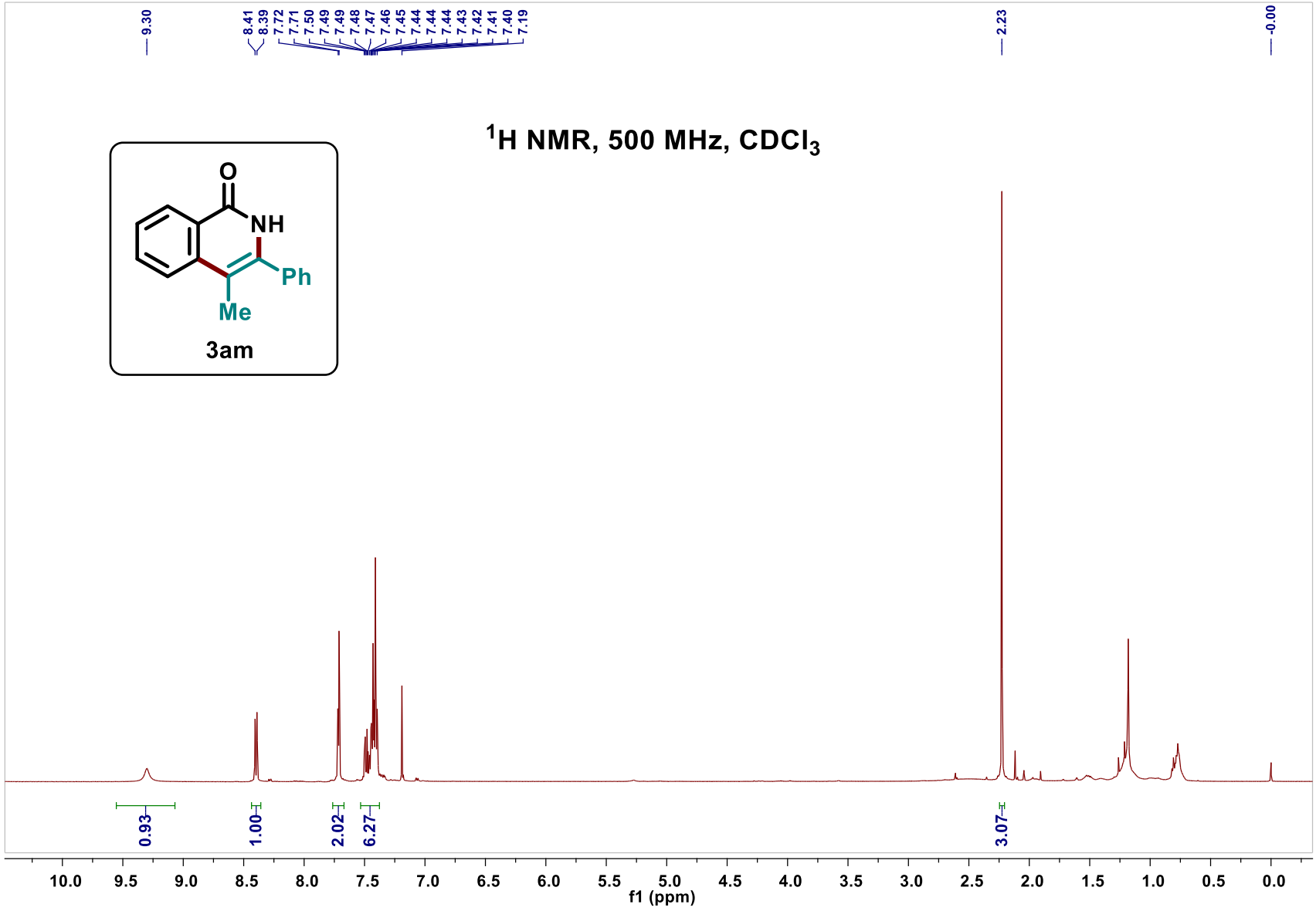

161.50
137.73
137.46
137.45
136.13
132.82
132.67
132.31
131.29
130.45
130.02
129.70
129.49
128.57
128.30
127.27
126.83
126.55
125.12
124.71
114.47
40.02
39.85
39.69
39.52
39.35
39.19
39.02
O
NH
Cl
Cl
3aj
13C NMR, 125 MHz, DMSO-D6
137.8
137.6
137.4
f1 (ppm)
133
132
131
130
129
128
127
126
125
f1 (ppm)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

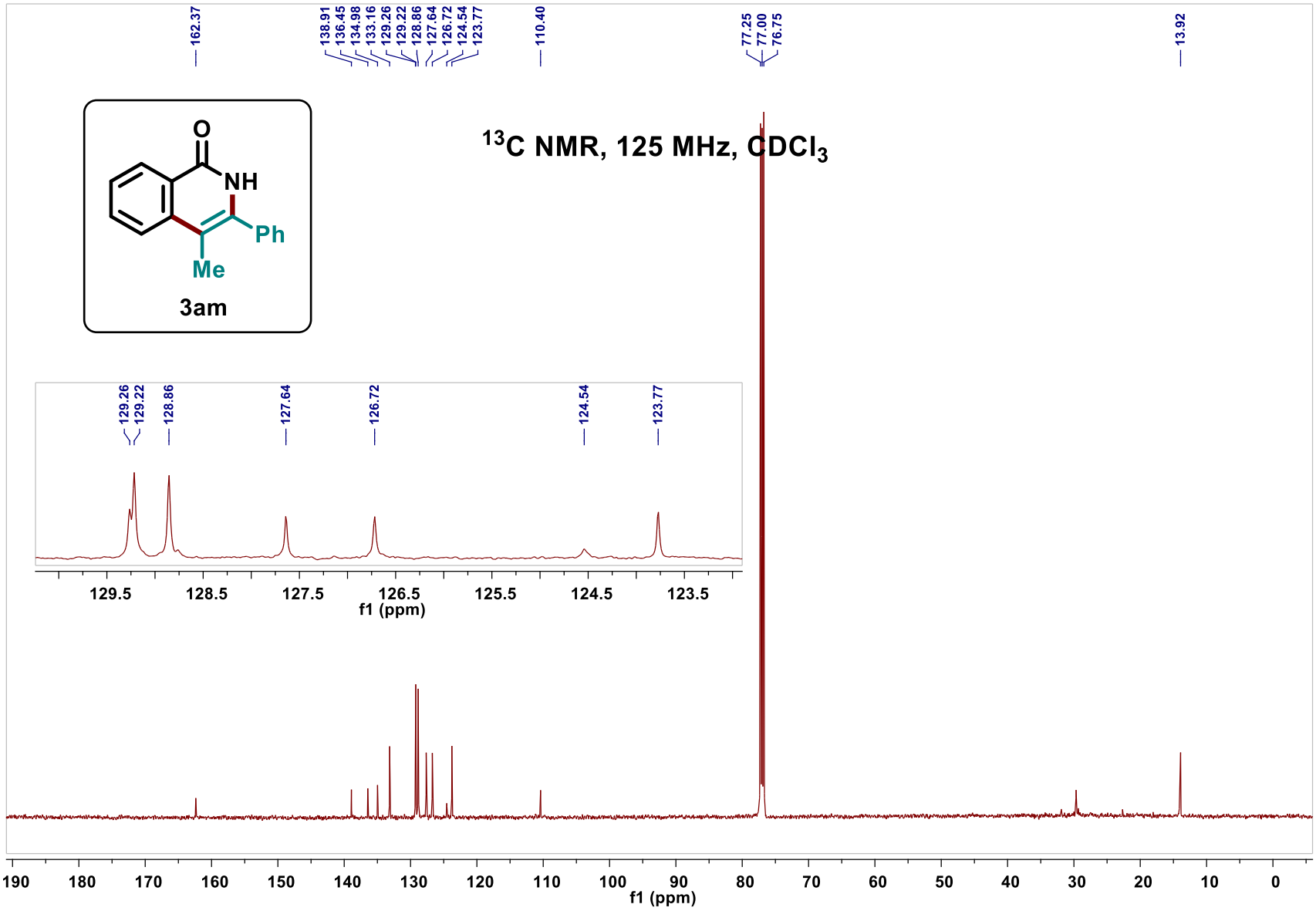

11.57
8.28 8.28 8.27 7.74 7.74 7.73 7.69 7.69 7.67 7.66 7.66 7.63 7.63 7.62 7.59 7.58 7.58 7.53 7.52 7.50 7.21 7.21 7.20 7.20 7.12 7.11 7.05 7.04 7.03
3.34
2.50 2.50 2.50
1H NMR, 500 MHz, DMSO-D6
O
NH
S
S
3ak
1.00 0.98 1.12 1.07 0.97 1.11 2.05 1.02 1.06
8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0
f1 (ppm)
* CHCl3
1.08 1.00 0.98 1.12 1.07 0.97 1.11 2.05 1.02 1.06
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 0.0
f1 (ppm)


161.62
138.60
135.76
134.70
134.12
132.81
131.24
129.89
129.28
128.64
127.72
126.59
126.35
125.02
124.57
107.06
40.02
39.85
39.69
39.52
39.35
39.19
39.02
^{13}C NMR, 125 MHz, DMSO-D$_6$
O
NH
S
S
3ak
f1 (ppm)
* CHCl$_3$
*
f1 (ppm)


11.73
8.48
8.47
7.69
7.68
7.68
7.67
7.67
7.66
7.65
7.47
7.46
7.45
7.45
7.44
7.44
7.43
7.42
7.41
7.26
2.72
2.72
2.71
2.70
2.70
2.69
2.69
1.77
1.76
1.74
1.61
1.07
1.06
1.05
1.03
O
NH
3al
1H NMR, 500 MHz, CDCl3
1.00
1.00
2.02
1.02
4.04
2.05
2.08
6.15
12.5
11.5
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


163.93
138.46
138.41
132.19
127.56
125.15
124.99
122.96
112.90
77.25
77.00
76.75
32.84
28.54
23.55
22.84
14.29
14.00
13C NMR, 125 MHz, CDCl3
O
NH
3al
f1 (ppm)
f1 (ppm)


9.30
8.41
8.39
7.72
7.71
7.50
7.49
7.49
7.48
7.47
7.46
7.45
7.44
7.44
7.44
7.43
7.42
7.41
7.40
7.19
2.23
-0.00
1H NMR, 500 MHz, CDCl3
O
NH
Ph
Me
3am
0.93
1.00
2.02
6.27
3.07
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
0.0
f1 (ppm)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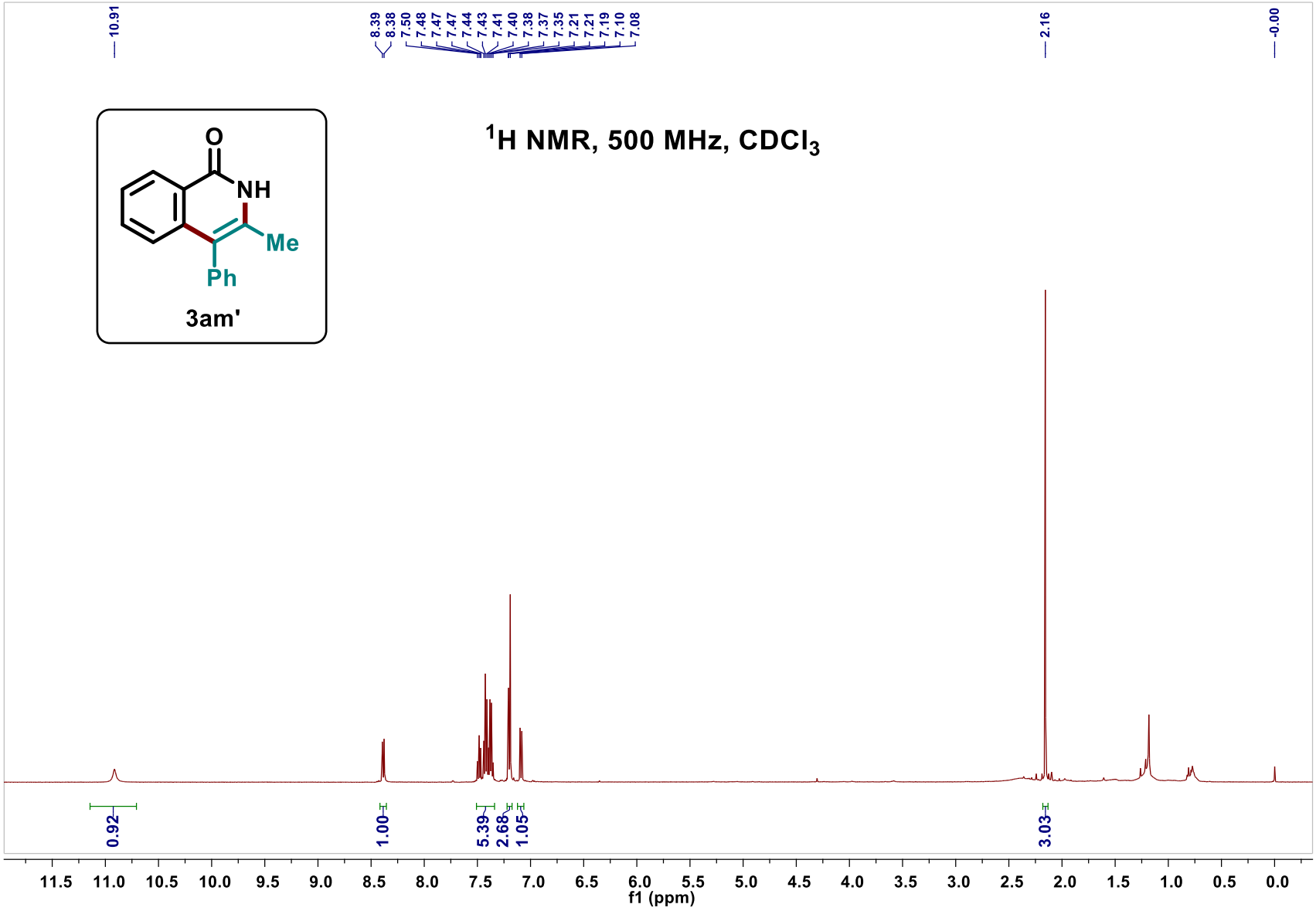
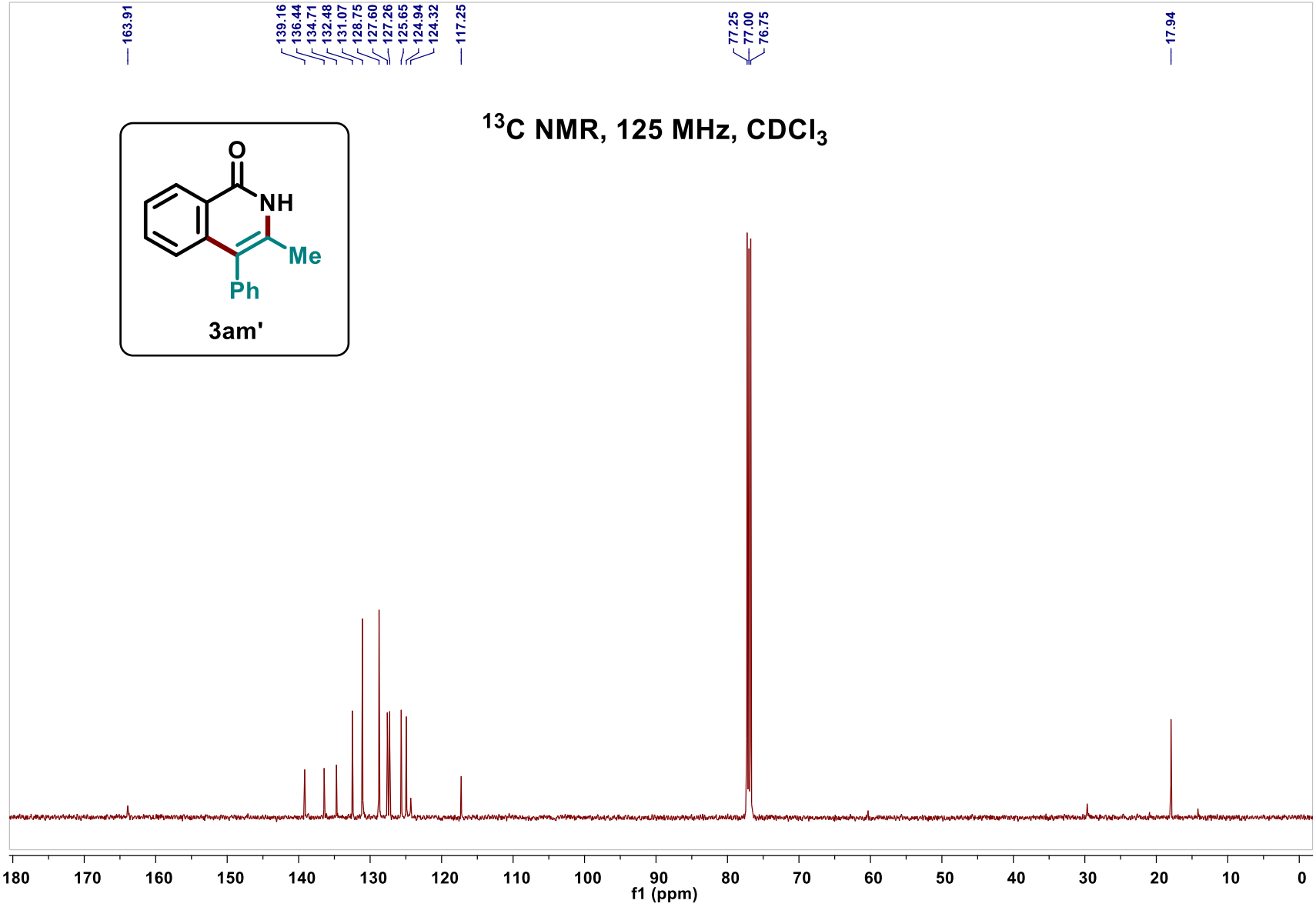
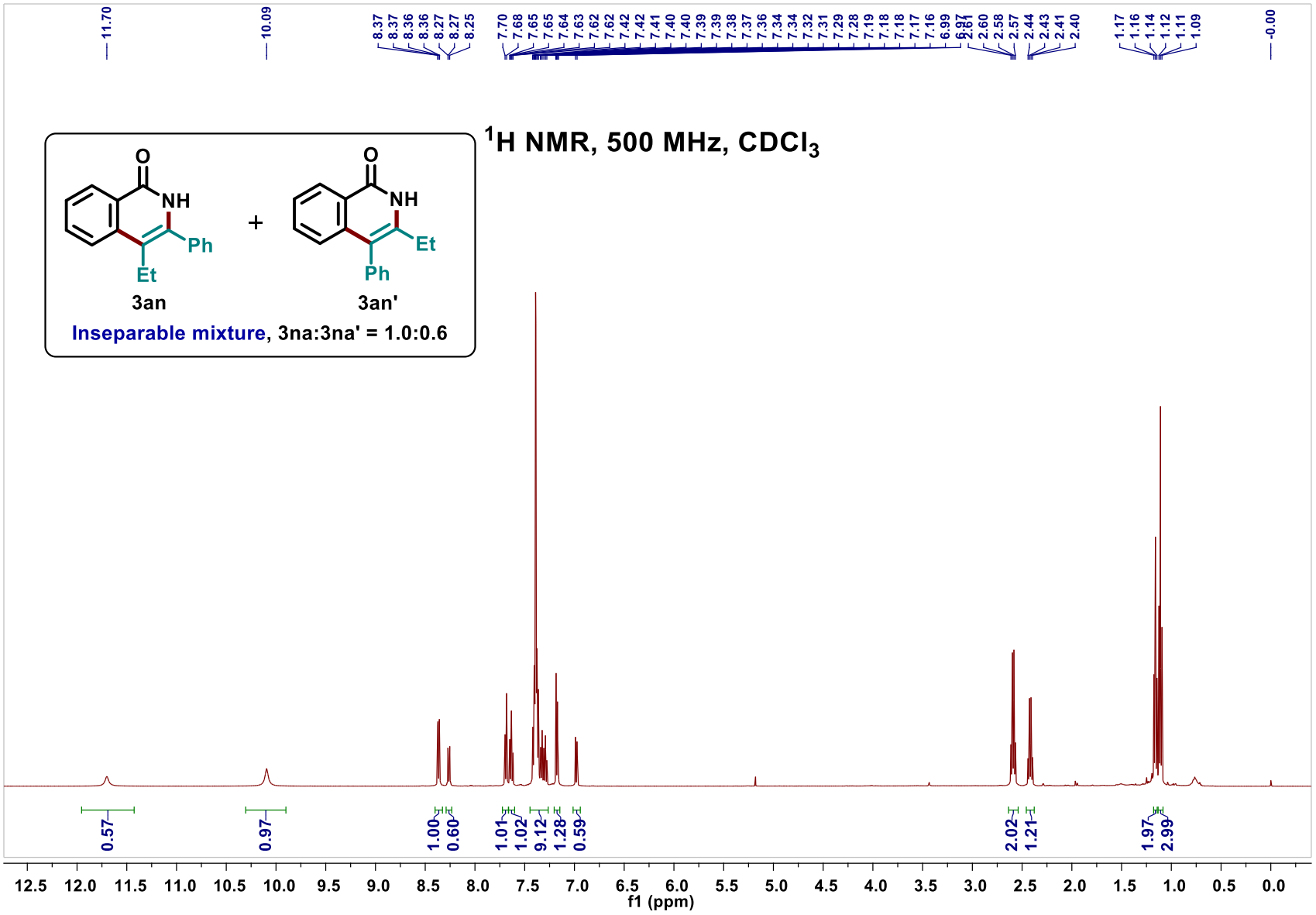
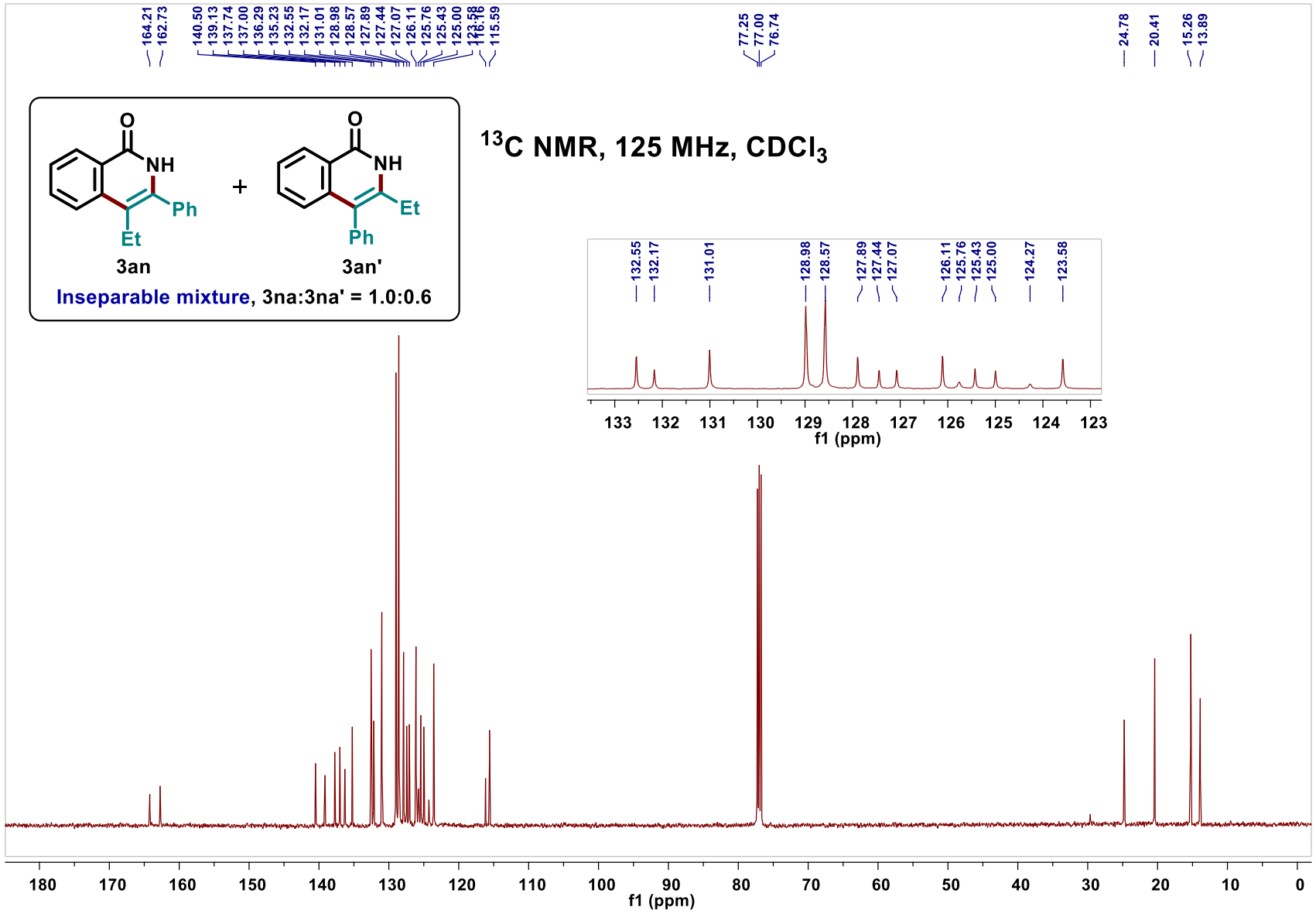
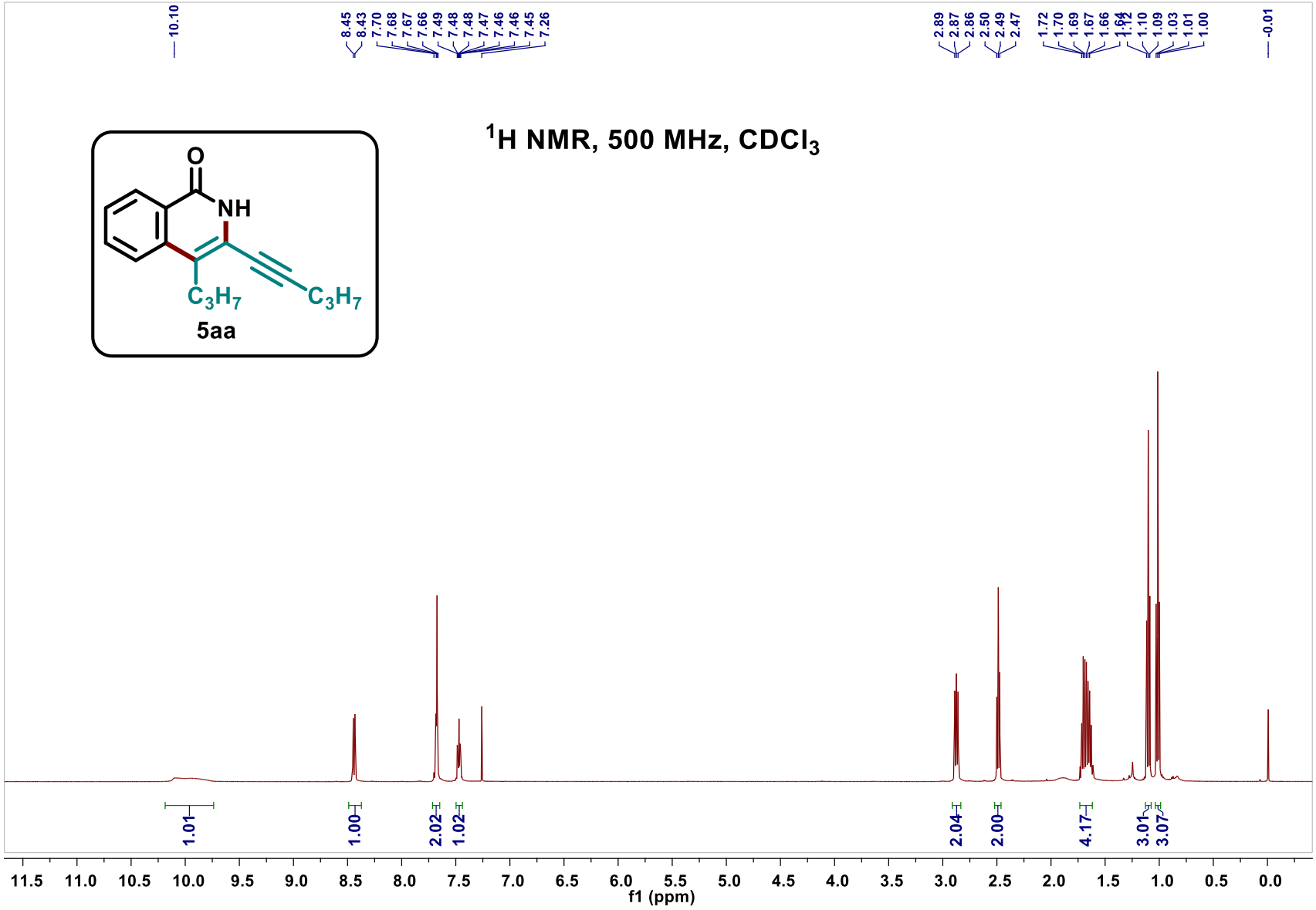
138.91
136.45
134.98
133.16
129.26
129.22
128.86
127.64
126.72
124.54
123.77
110.40
77.25
77.00
76.75
13.92
O
NH
Ph
Me
3am
^{13}C NMR, 125 MHz, CDCl$_3$
129.26
129.22
128.86
127.64
126.72
124.54
123.77
129.5
128.5
127.5
126.5
125.5
124.5
123.5
f1 (ppm)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

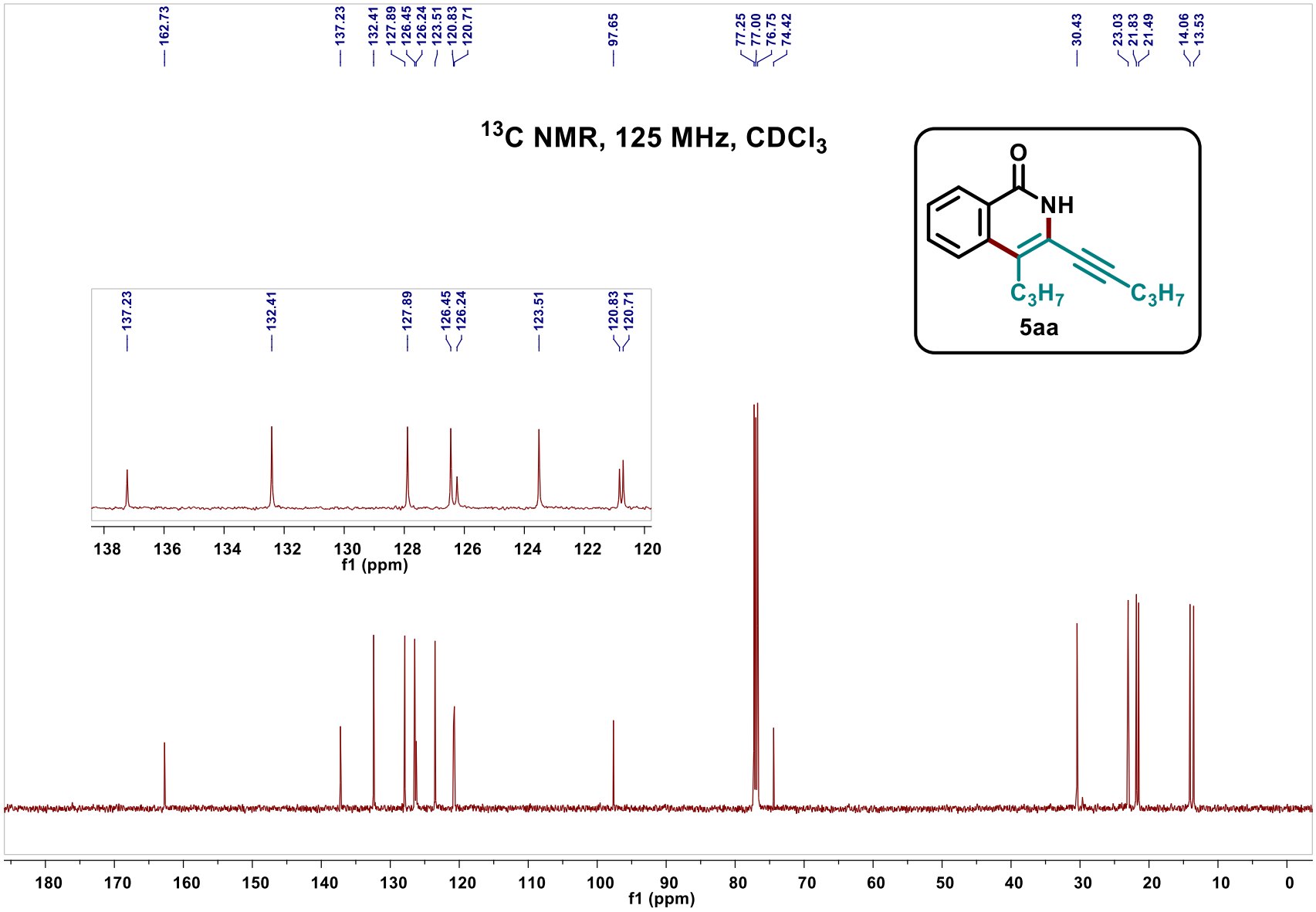

10.91
8.39
8.38
7.50
7.48
7.47
7.47
7.44
7.43
7.41
7.40
7.38
7.37
7.35
7.21
7.21
7.19
7.10
7.08
2.16
-0.00
O
NH
Me
Ph
3am'
^{1}H NMR, 500 MHz, CDCl$_3$
0.92
1.00
5.39
2.68
1.05
3.03
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


163.91
139.16
136.44
134.71
132.48
131.07
128.75
127.60
127.26
125.65
124.94
124.32
117.25
77.25
77.00
76.75
17.94
13C NMR, 125 MHz, CDCl3
O
NH
Me
Ph
3am'
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


11.70
10.09
8.37 8.37 8.36 8.36 8.27 8.27 8.25
7.70 7.68 7.65 7.65 7.64 7.63 7.62 7.62 7.42 7.42 7.41 7.40 7.40 7.39 7.39 7.38 7.37 7.36 7.34 7.34 7.32 7.31 7.29 7.28 7.19 7.18 7.18 7.17 7.16 6.99
2.60 2.58 2.57 2.44 2.43 2.41 2.40
1.17 1.16 1.14 1.12 1.11 1.09
-0.00
1H NMR, 500 MHz, CDCl3
O
NH
Ph
Et
3an
+
O
NH
Et
Ph
3an'
Inseparable mixture, 3na:3na' = 1.0:0.6
0.57 0.97 1.00 0.60 1.01 1.02 9.12 1.28 0.59 2.02 1.21 1.97 2.99
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 0.0
f1 (ppm)


164.21
162.73
140.50
139.13
137.74
137.00
136.29
135.23
132.55
132.17
131.01
128.98
128.57
127.89
127.44
127.07
126.11
125.76
125.43
125.00
123.58
116.16
115.59
77.25
77.00
76.74
24.78
20.41
15.26
13.89
O
NH
Ph
Et
3an
+
O
NH
Et
Ph
3an'
Inseparable mixture, 3na:3na' = 1.0:0.6
13C NMR, 125 MHz, CDCl3
132.55
132.17
131.01
128.98
128.57
127.89
127.44
127.07
126.11
125.76
125.43
125.00
124.27
123.58
133 132 131 130 129 128 127 126 125 124 123
f1 (ppm)
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


10.10
8.45
8.43
7.70
7.68
7.67
7.66
7.49
7.48
7.48
7.47
7.46
7.46
7.45
7.26
2.89
2.87
2.86
2.50
2.49
2.47
1.72
1.70
1.69
1.67
1.66
1.64
1.62
1.10
1.09
1.03
1.01
1.00
-0.01
O
NH
C3H7
C3H7
5aa
1H NMR, 500 MHz, CDCl3
1.01
1.00
2.02
1.02
2.04
2.00
4.17
3.01
3.07
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
0.0
f1 (ppm)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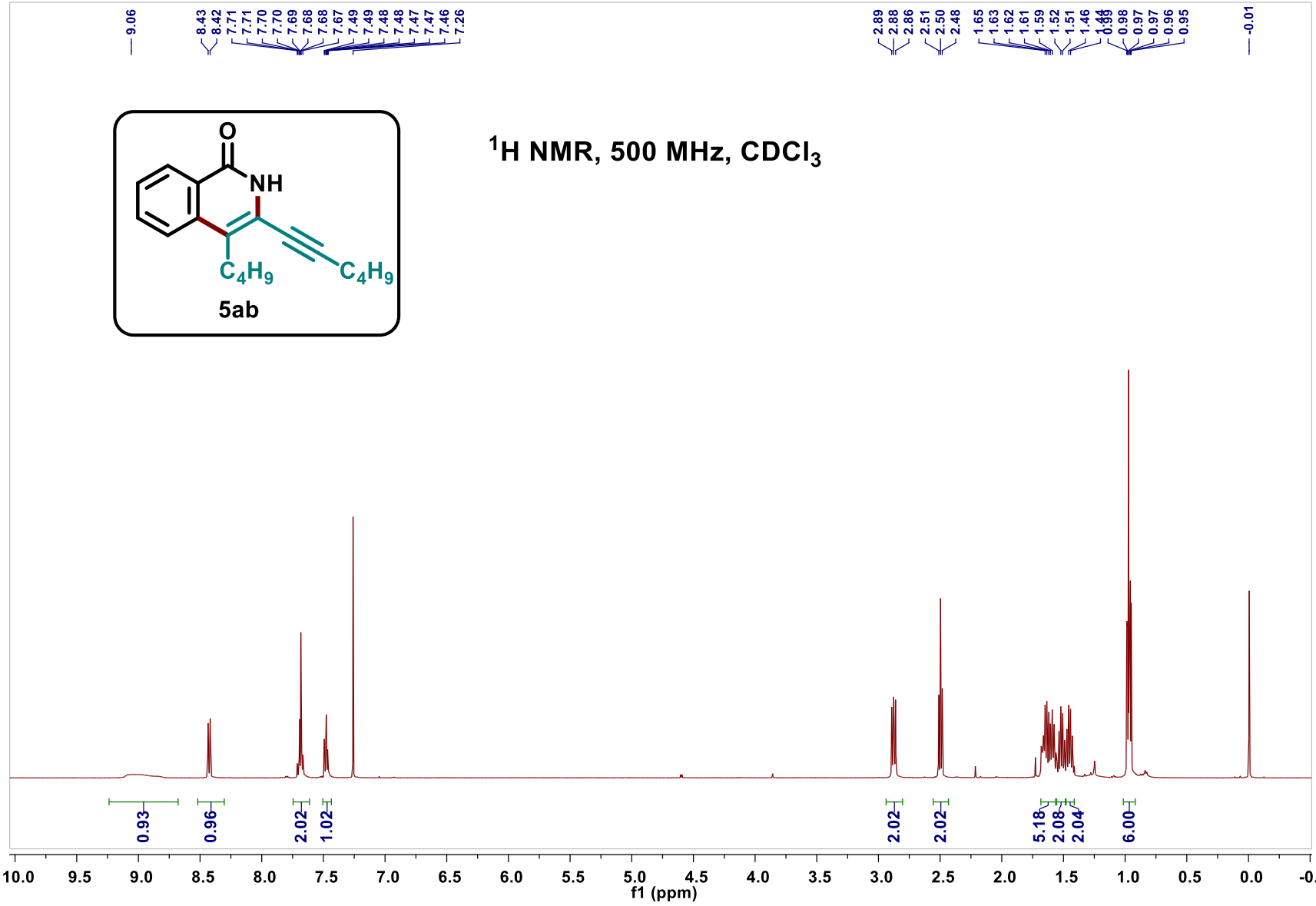
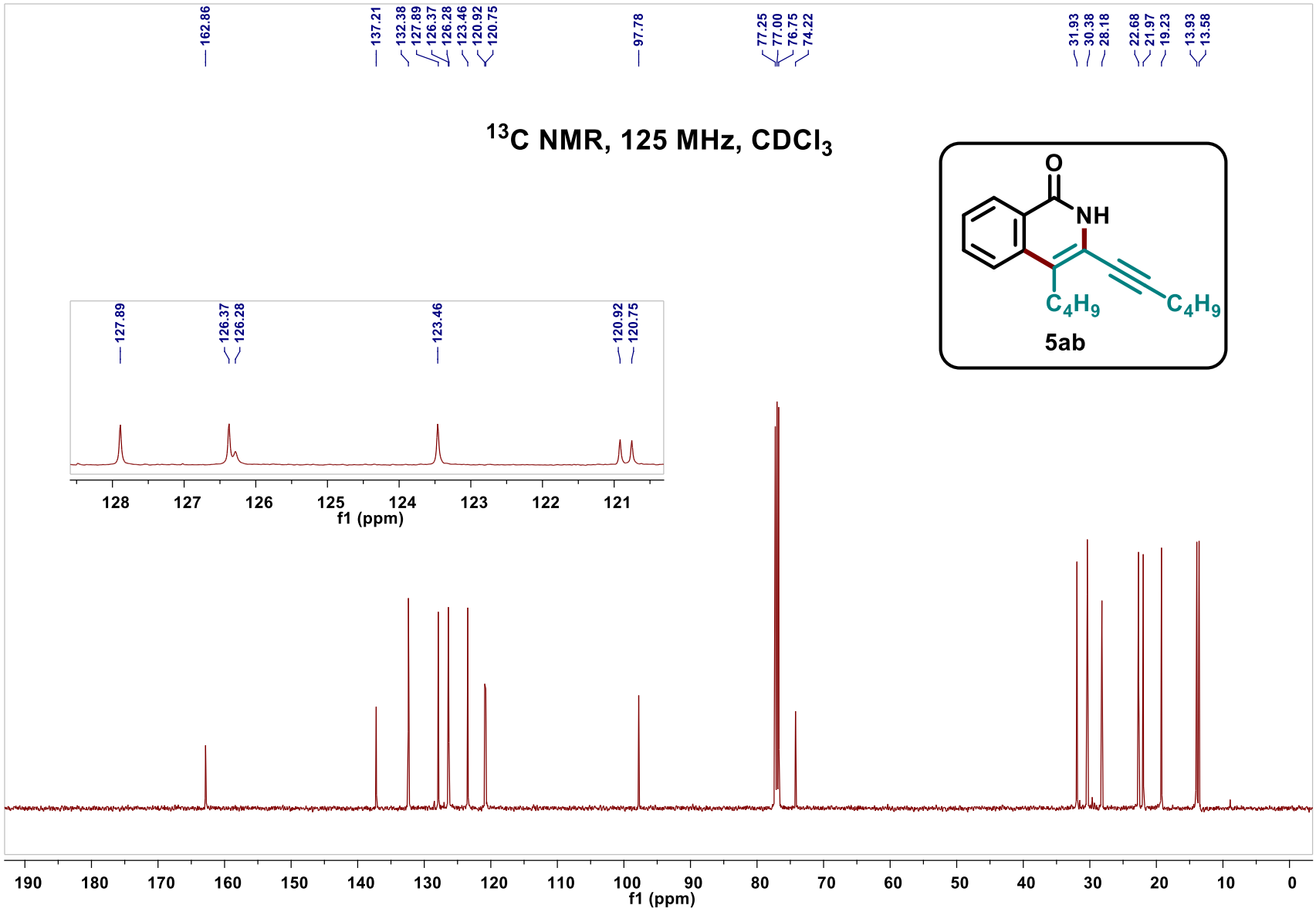
137.23
132.41
127.89
126.45
126.24
123.51
120.83
120.71
97.65
77.25
77.00
76.75
74.42
30.43
23.03
21.83
21.49
14.06
13.53
^{13}C NMR, 125 MHz, $CDCl_3$
O
NH
C_3H_7
C_3H_7
5aa
f1 (ppm)
f1 (ppm)


9.06
8.43
8.42
7.71
7.71
7.70
7.70
7.69
7.68
7.68
7.67
7.49
7.49
7.48
7.48
7.47
7.47
7.46
7.26
2.89
2.88
2.86
2.51
2.50
2.48
1.65
1.63
1.62
1.61
1.59
1.52
1.51
1.46
0.98
0.97
0.97
0.96
0.95
-0.01
O
NH
C4H9
C4H9
5ab
1H NMR, 500 MHz, CDCl3
0.93
0.96
2.02
1.02
2.02
2.02
5.18
2.08
2.04
6.00
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
0.0
f1 (ppm)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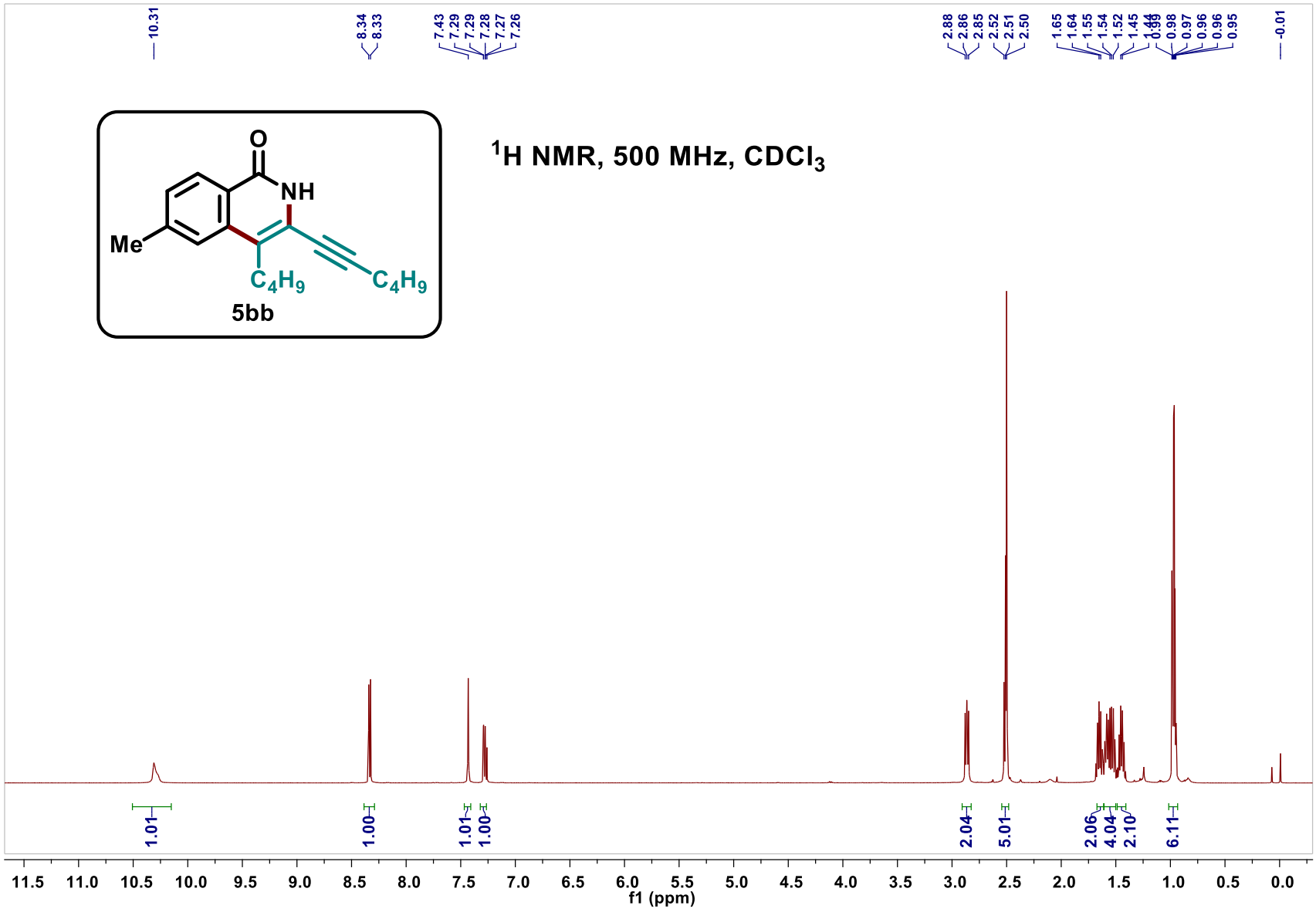
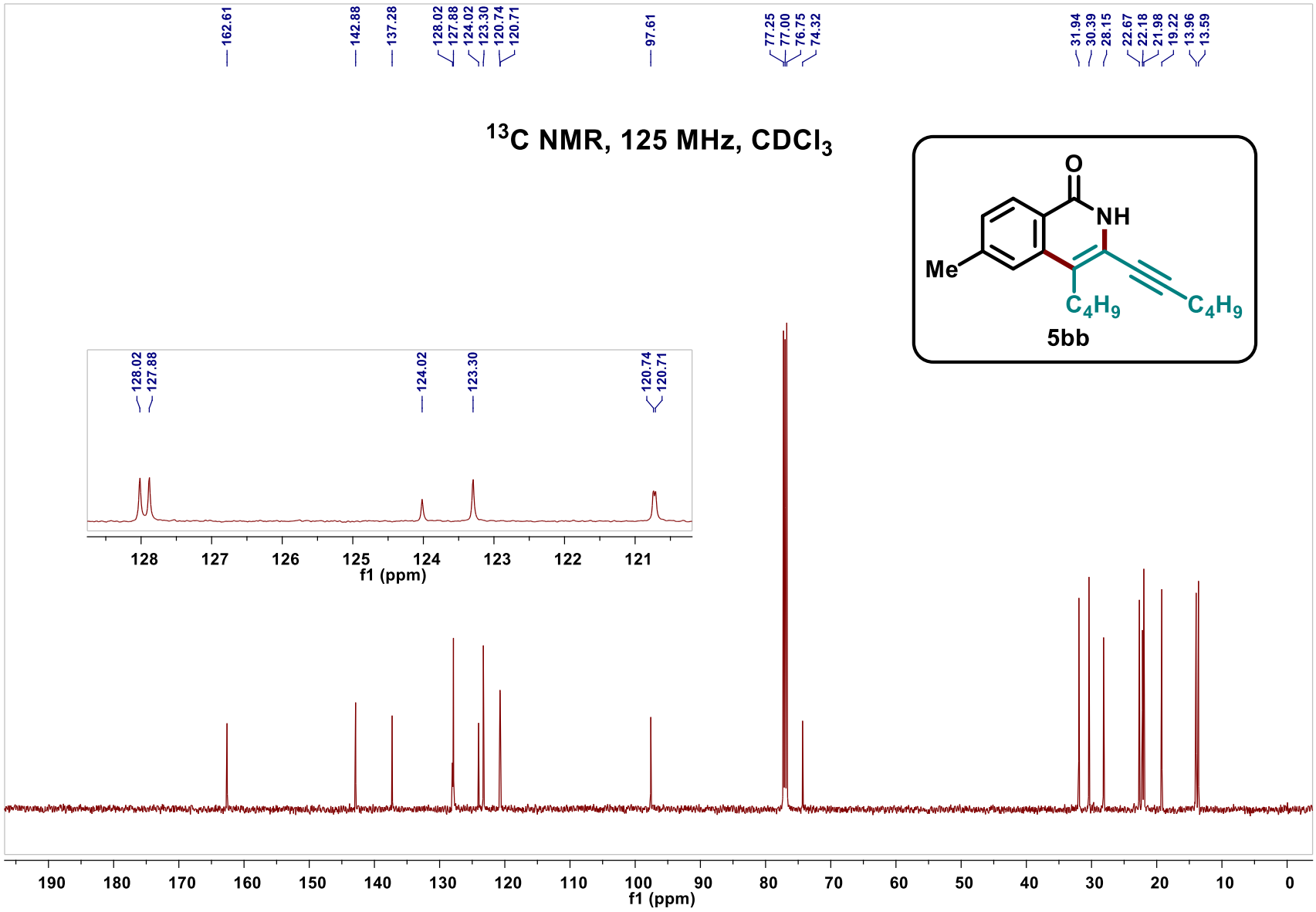
127.89
126.37
126.28
123.46
120.92
120.75
97.78
77.25
77.00
76.75
74.22
31.93
30.38
28.18
22.68
21.97
19.23
13.93
13.58
13C NMR, 125 MHz, CDCl3
O
NH
C4H9
C4H9
5ab
127.89
126.37
126.28
123.46
120.92
120.75
128
127
126
125
124
123
122
121
f1 (ppm)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


10.31
8.34
8.33
7.43
7.29
7.29
7.28
7.27
7.26
2.88
2.86
2.85
2.52
2.51
2.50
1.65
1.64
1.55
1.54
1.52
1.45
0.98
0.97
0.96
0.96
0.95
-0.01
O
NH
Me
C4H9
C4H9
5bb
1H NMR, 500 MHz, CDCl3
1.01
1.00
1.01
1.00
2.04
5.01
2.06
4.04
2.10
6.11
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 0.0
f1 (ppm)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142.88
137.28
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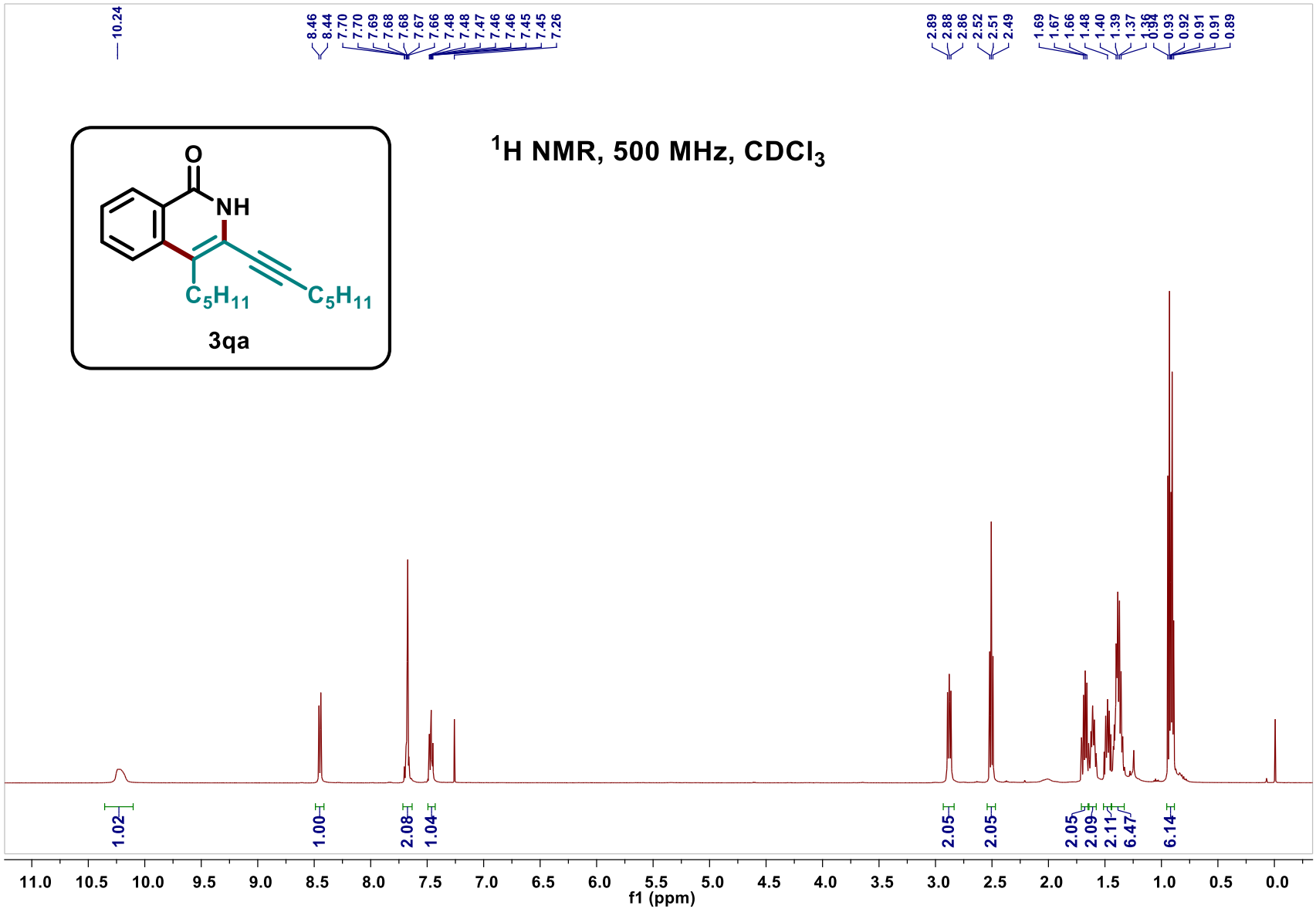
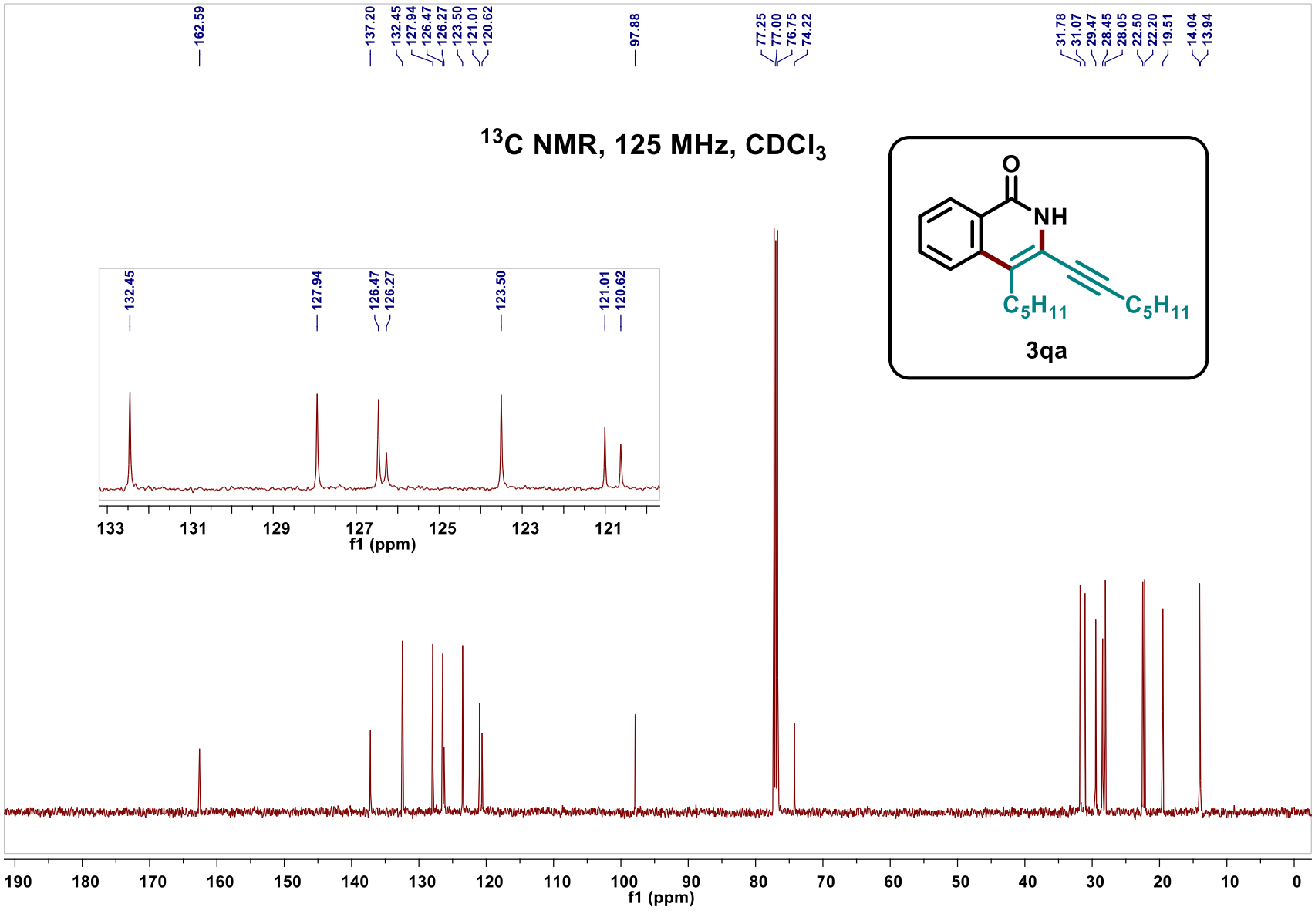
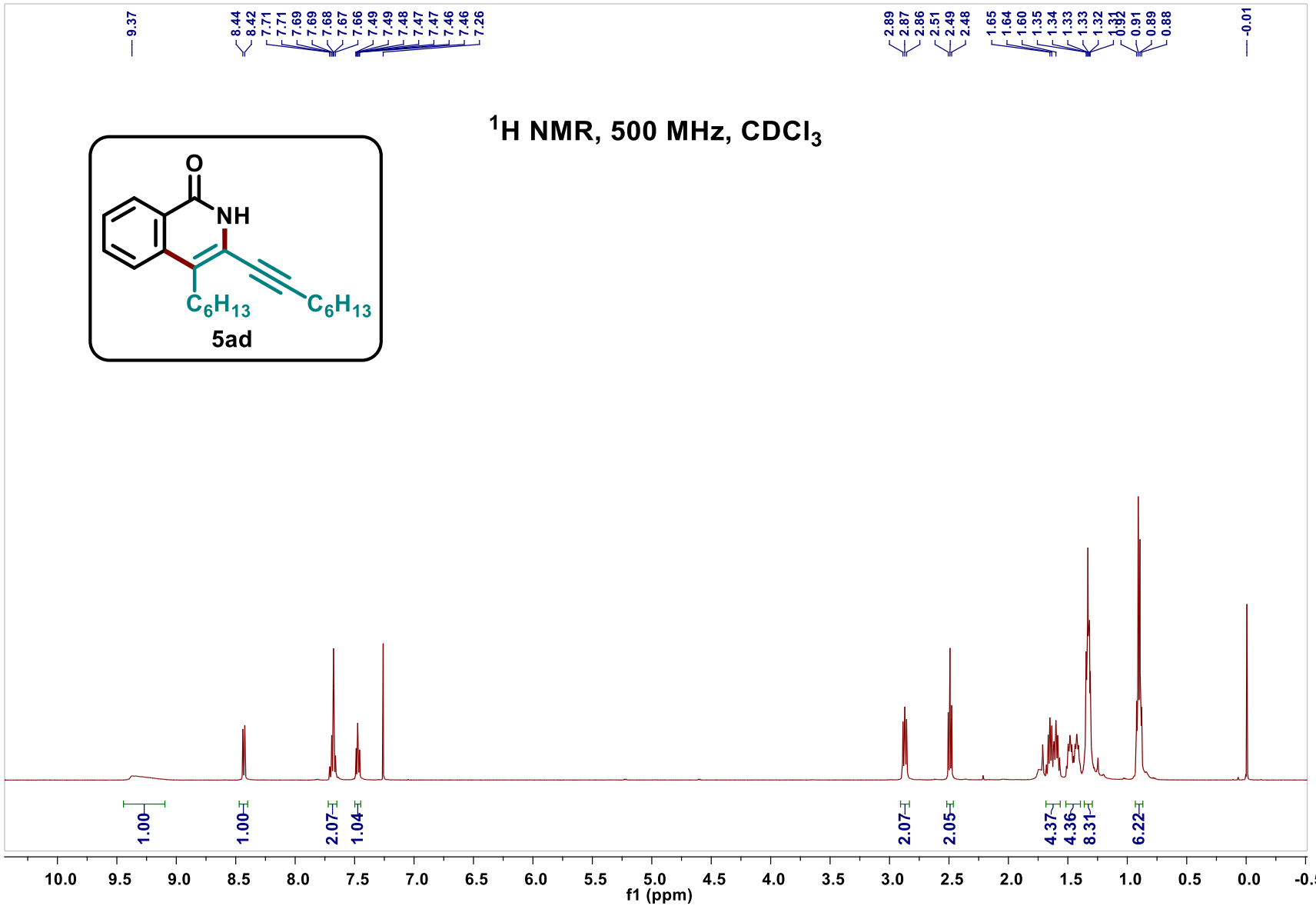
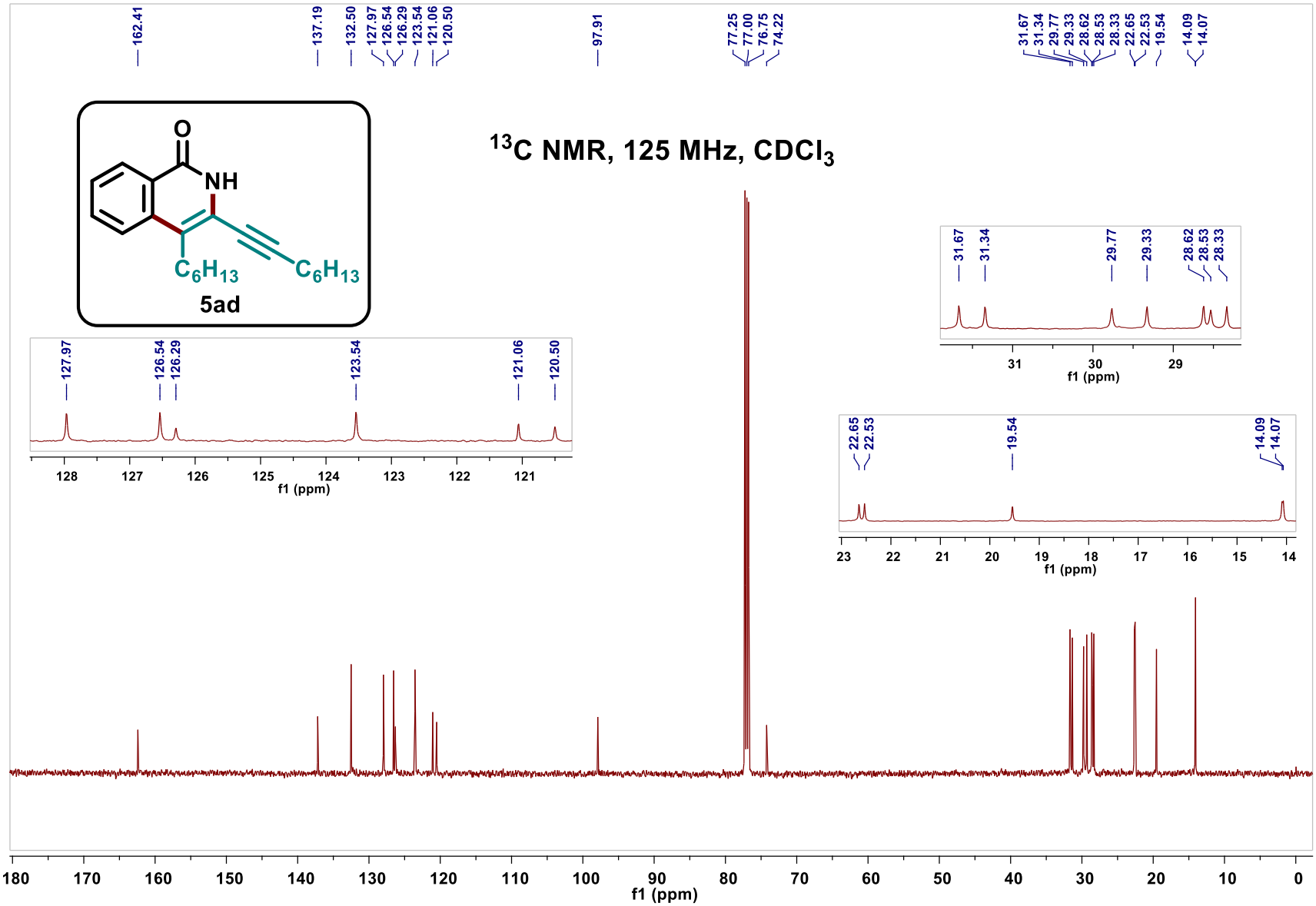
124.02
123.30
120.74
120.71
97.61
77.25
77.00
76.75
74.32
31.94
30.39
28.15
22.67
22.18
21.98
19.22
13.96
13.59
13C NMR, 125 MHz, CDCl3
O
NH
Me
C4H9
C4H9
5bb
f1 (ppm)
f1 (ppm)


10.24
8.46
8.44
7.70
7.70
7.69
7.68
7.68
7.67
7.66
7.48
7.48
7.47
7.46
7.46
7.45
7.45
7.26
2.89
2.88
2.86
2.52
2.51
2.49
1.69
1.67
1.66
1.48
1.40
1.39
1.37
0.94
0.93
0.92
0.91
0.91
0.89
O
NH
C5H11
C5H11
3qa
1H NMR, 500 MHz, CDCl3
1.02
1.00
2.08
1.04
2.05
2.05
2.05
2.09
2.11
6.47
6.14
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
0.0
f1 (ppm)


162.59
137.20
132.45
127.94
126.47
126.27
123.50
121.01
120.62
97.88
77.25
77.00
76.75
74.22
31.78
31.07
29.47
28.45
28.05
22.50
22.20
19.51
14.04
13.94
13C NMR, 125 MHz, CDCl3
O
NH
C5H11
C5H11
3qa
f1 (ppm)
f1 (ppm)

¥


9.37
8.44
8.42
7.71
7.71
7.69
7.69
7.68
7.67
7.66
7.49
7.49
7.48
7.47
7.47
7.46
7.46
7.26
2.89
2.87
2.86
2.51
2.49
2.48
1.65
1.64
1.60
1.35
1.34
1.33
1.33
1.32
1.31
0.92
0.91
0.89
0.88
-0.01
1H NMR, 500 MHz, CDCl3
O
NH
C6H13
C6H13
5ad
1.00
1.00
2.07
1.04
2.07
2.05
4.37
4.36
8.31
6.22
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
0.0
f1 (ppm)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137.19
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126.54
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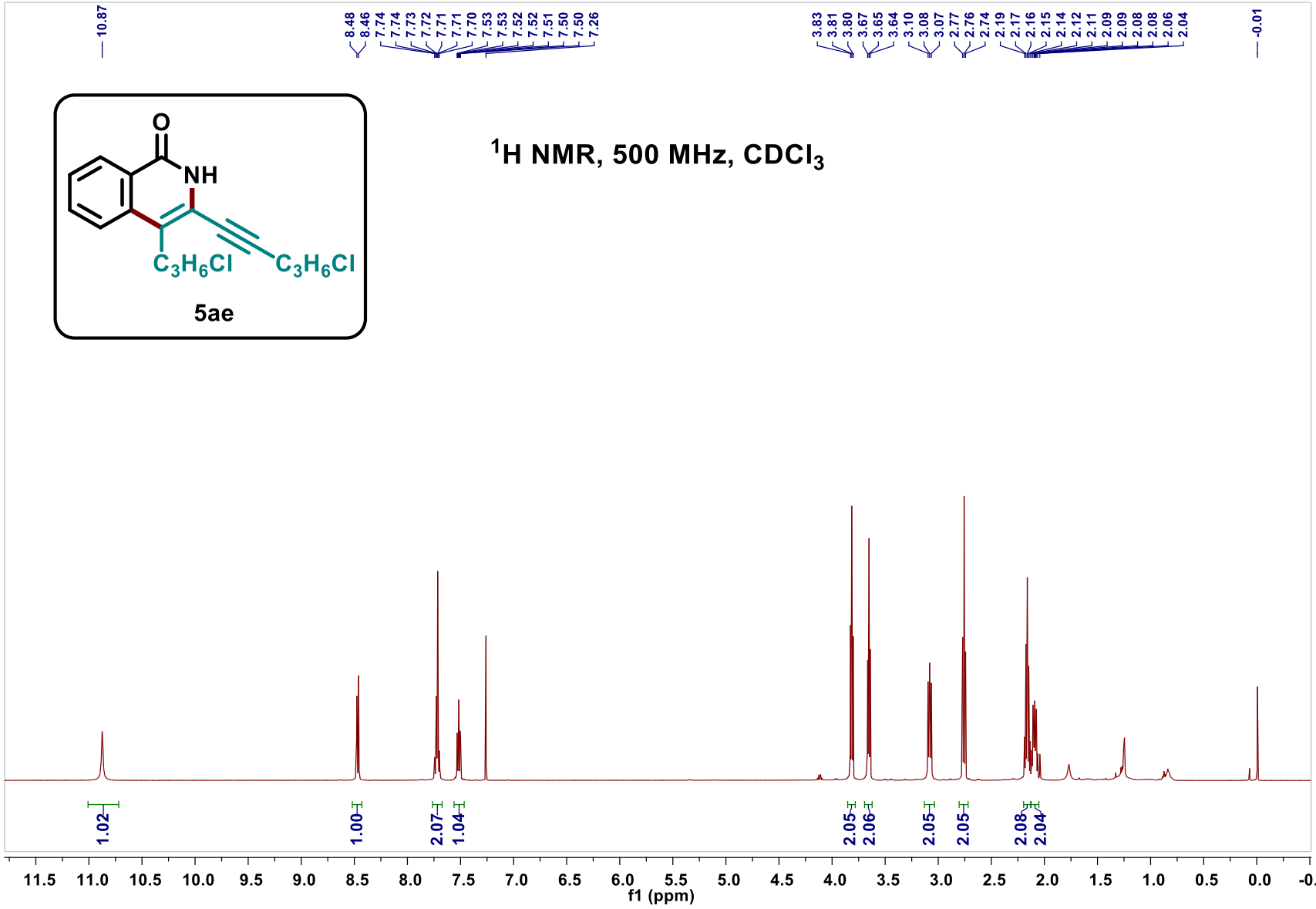
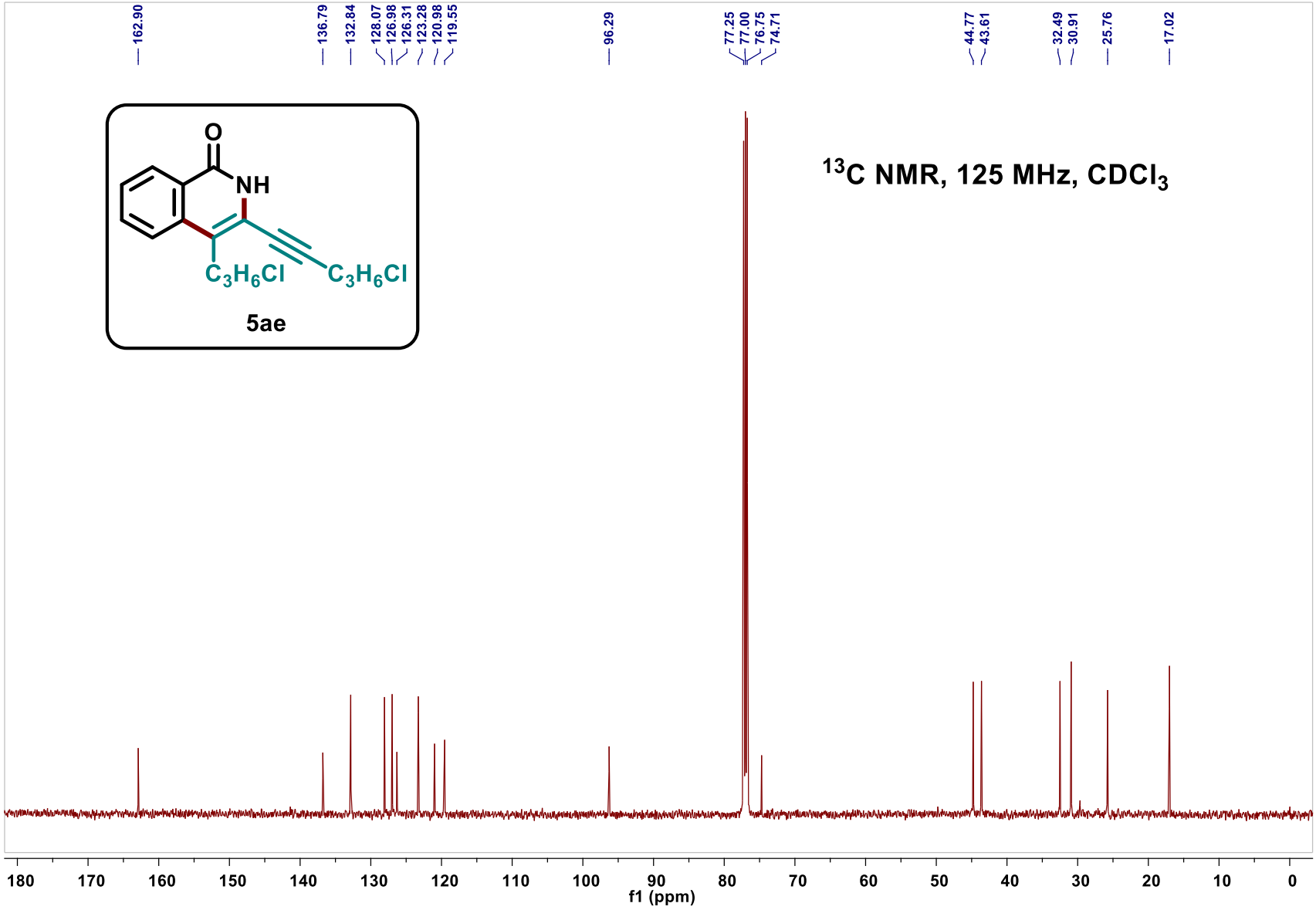
123.54
121.06
120.50
97.91
77.25
77.00
76.75
74.22
31.67
31.34
29.77
29.33
28.62
28.53
28.33
22.65
22.53
19.54
14.09
14.07
O
NH
C6H13
C6H13
5ad
13C NMR, 125 MHz, CDCl3
f1 (ppm)


10.87
8.48
8.46
7.74
7.74
7.73
7.72
7.71
7.71
7.70
7.53
7.52
7.52
7.51
7.50
7.50
7.26
3.83
3.81
3.80
3.67
3.65
3.64
3.10
3.08
3.07
2.77
2.76
2.74
2.19
2.17
2.16
2.15
2.14
2.12
2.11
2.09
2.09
2.08
2.08
2.06
2.04
-0.01
O
NH
C3H6Cl
C3H6Cl
5ae
1H NMR, 500 MHz, CDCl3
1.02
1.00
2.07
1.04
2.05
2.06
2.05
2.05
2.08
2.04
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
0.0
f1 (ppm)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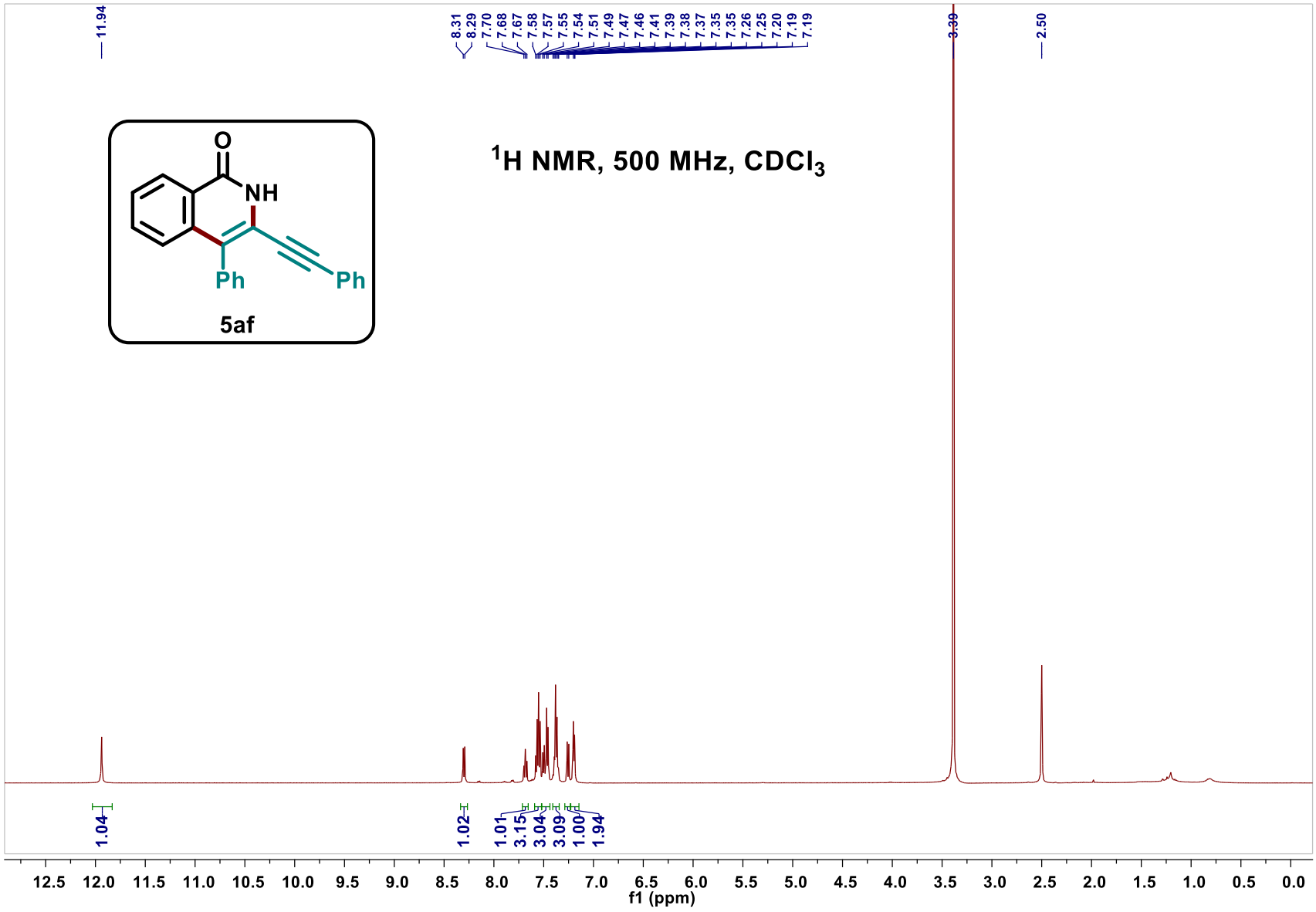
136.79
132.84
128.07
126.98
126.31
123.28
120.98
119.55
96.29
77.25
77.00
76.75
74.71
44.77
43.61
32.49
30.91
25.76
17.02
O
NH
C3H6Cl
C3H6Cl
5ae
13C NMR, 125 MHz, CDCl3
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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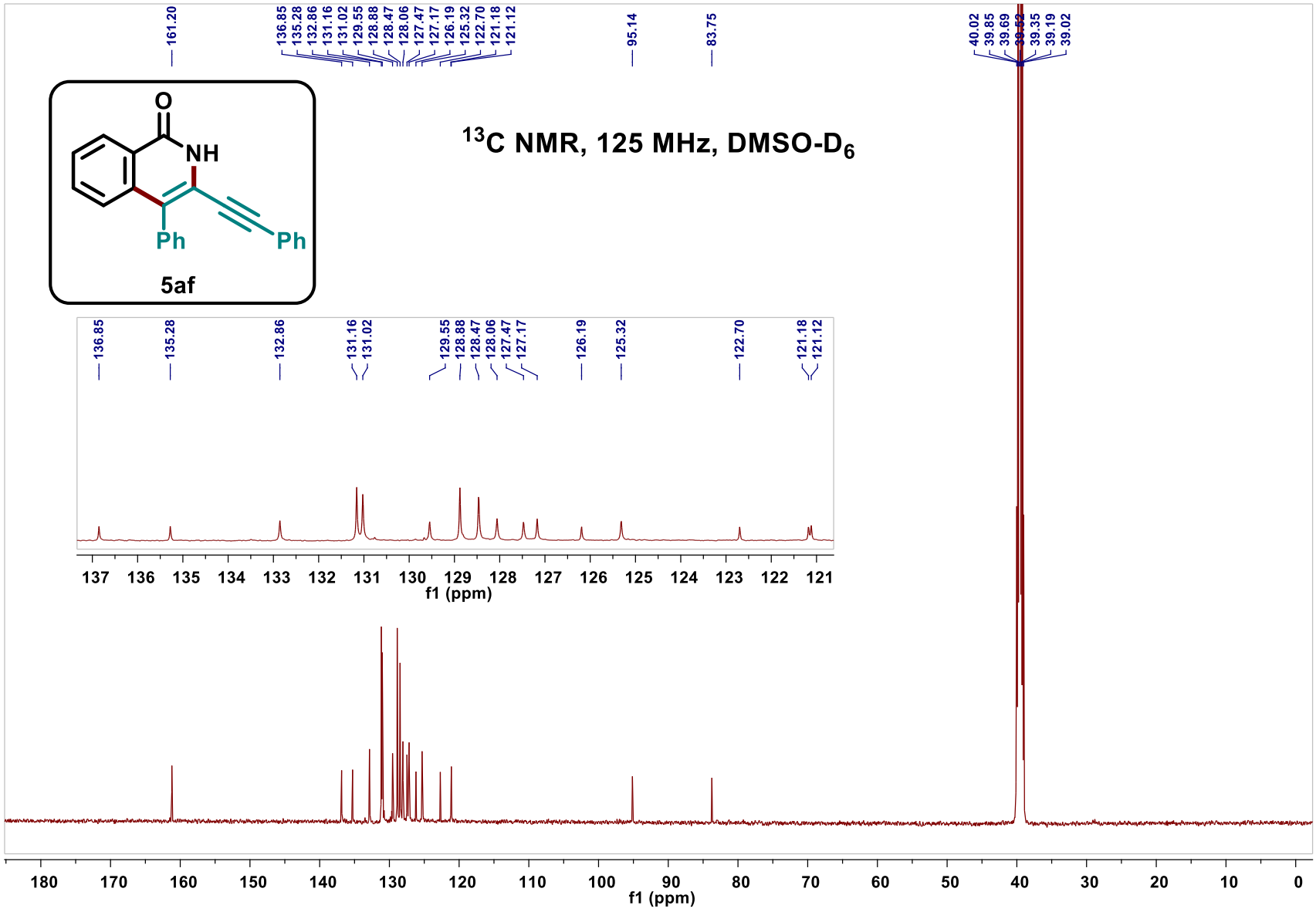
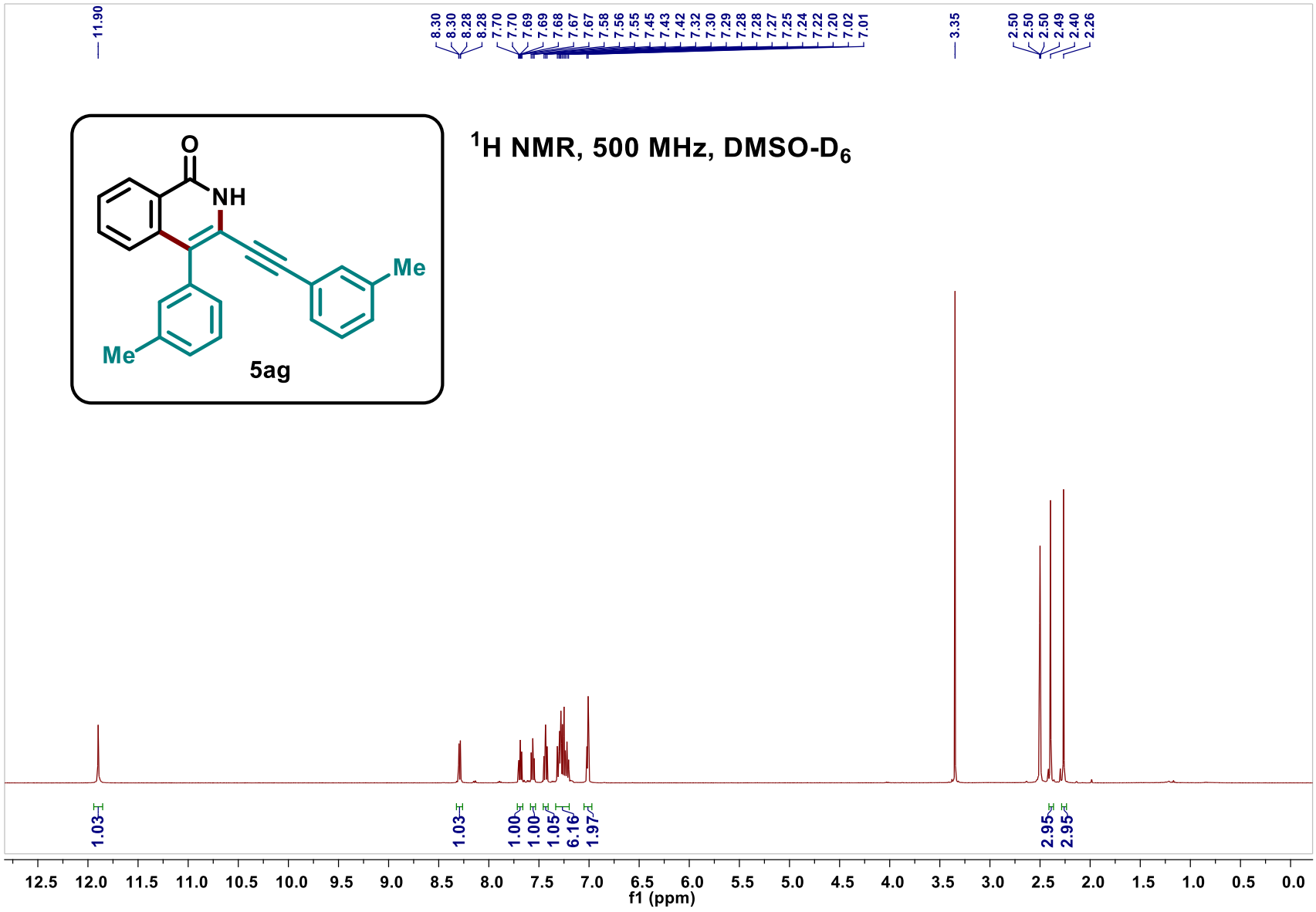
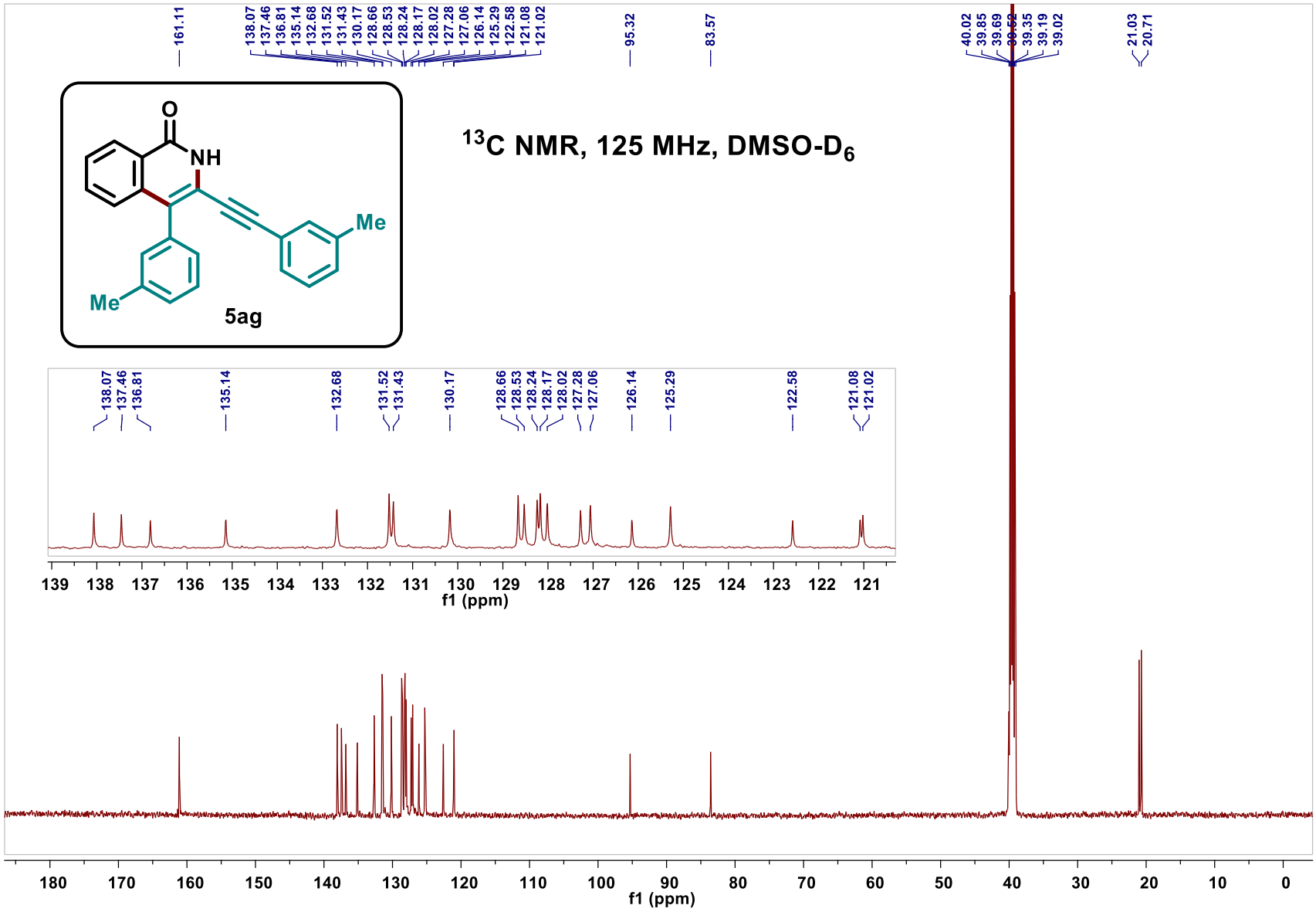
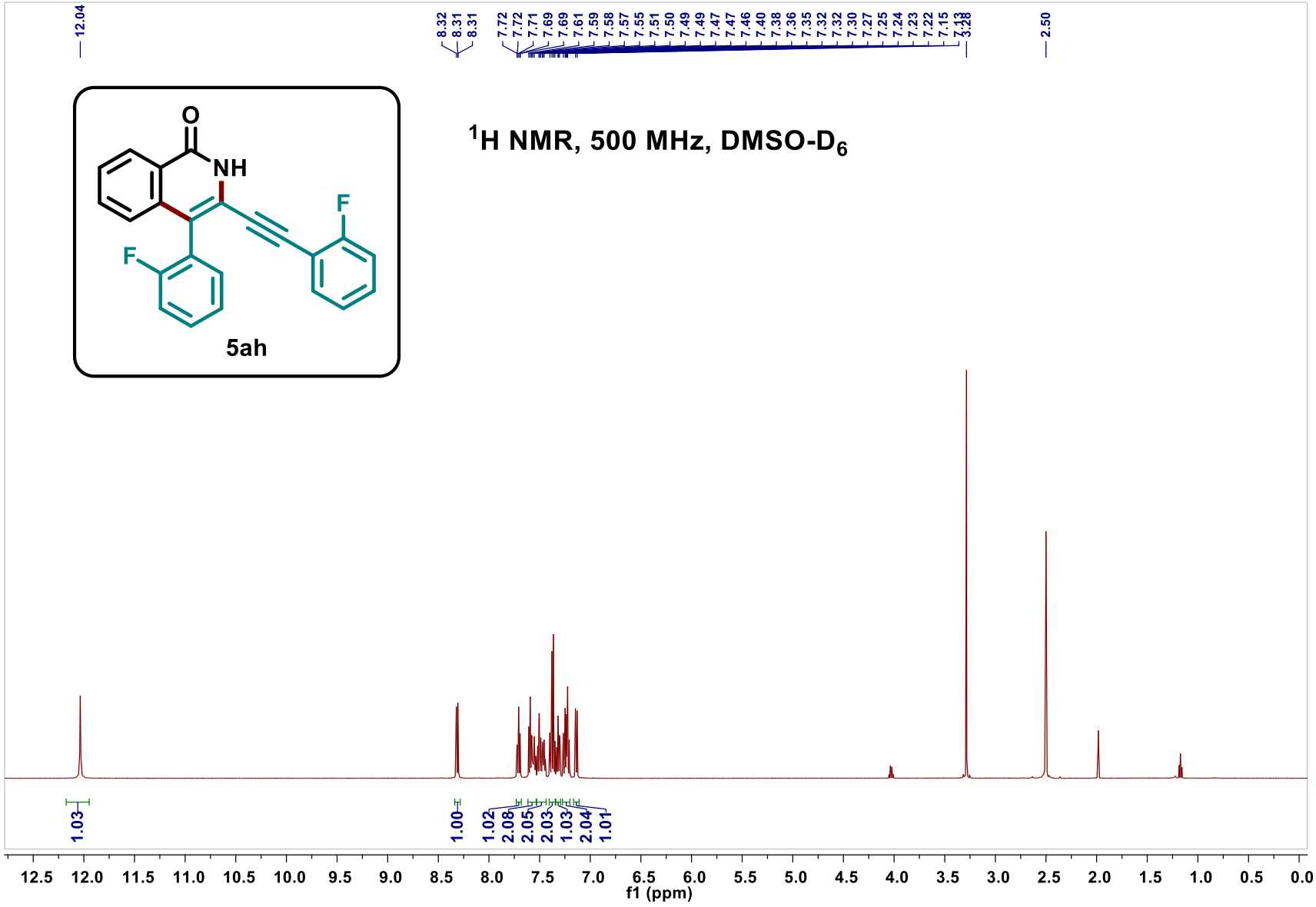
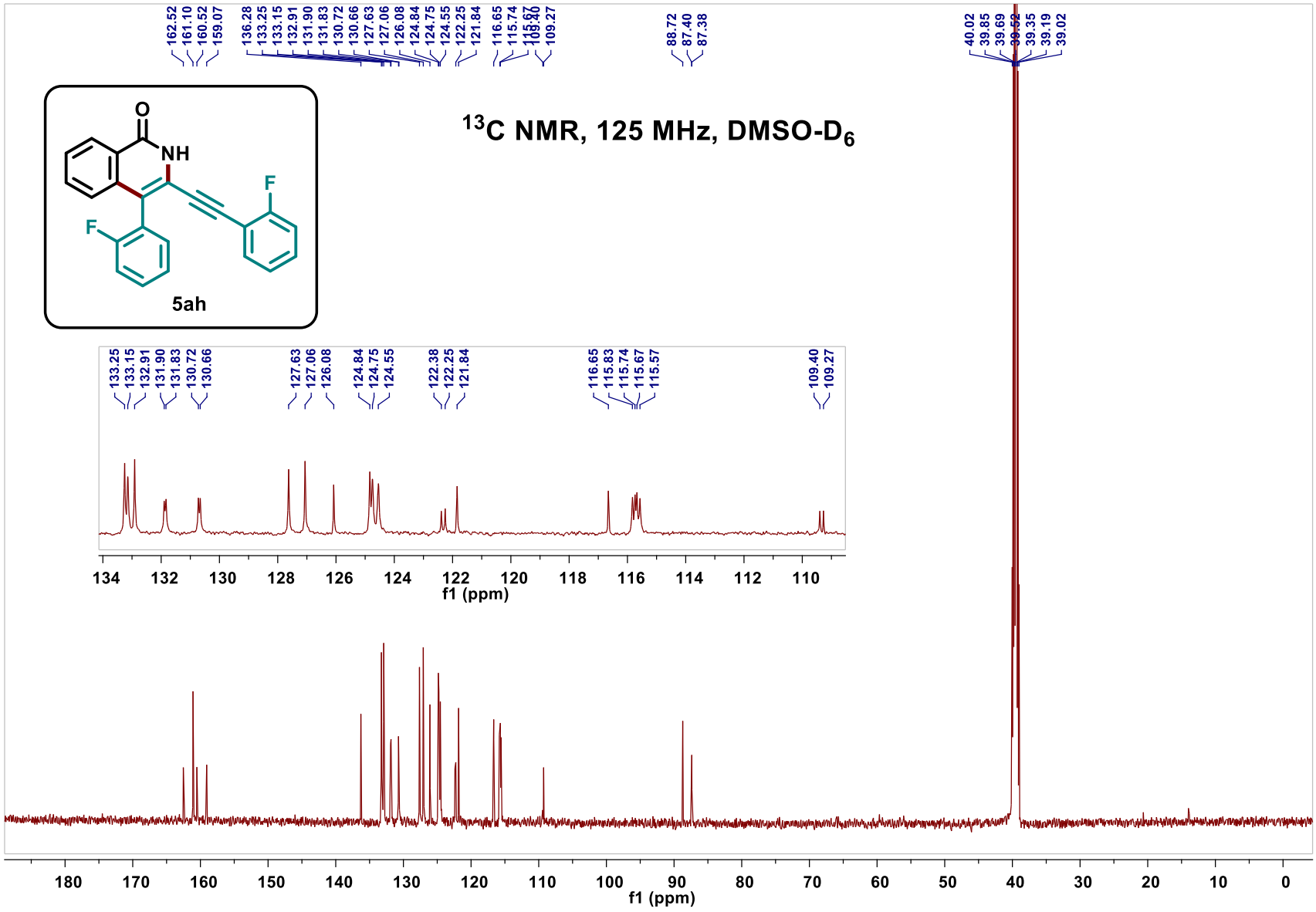
8.31
8.29
7.70
7.68
7.67
7.58
7.57
7.55
7.54
7.51
7.49
7.47
7.46
7.41
7.39
7.38
7.37
7.35
7.35
7.26
7.25
7.20
7.19
7.19
3.99
2.50
O
NH
Ph
Ph
5af
1H NMR, 500 MHz, CDCl3
1.04
1.02
1.01
3.15
3.04
3.09
1.00
1.94
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


161.20
136.85
135.28
132.86
131.16
131.02
129.55
128.88
128.47
128.06
127.47
127.17
126.19
125.32
122.70
121.18
121.12
95.14
83.75
40.02
39.85
39.69
39.52
39.35
39.19
39.02
O
NH
Ph
Ph
5af
^{13}C NMR, 125 MHz, DMSO-D$_6$
f1 (ppm)
f1 (ppm)


11.90
8.30
8.30
8.28
8.28
7.70
7.70
7.69
7.69
7.68
7.67
7.67
7.58
7.56
7.55
7.45
7.43
7.42
7.32
7.30
7.29
7.28
7.28
7.27
7.25
7.24
7.22
7.20
7.02
7.01
3.35
2.50
2.50
2.50
2.49
2.40
2.26
O
NH
Me
Me
5ag
1H NMR, 500 MHz, DMSO-D6
1.03
1.03
1.00
1.00
1.05
6.16
1.97
2.95
2.95
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 0.0
f1 (ppm)


161.11
138.07
137.46
136.81
135.14
132.68
131.52
131.43
130.17
128.66
128.53
128.24
128.17
128.02
127.28
127.06
126.14
125.29
122.58
121.08
121.02
95.32
83.57
40.02
39.85
39.69
39.52
39.35
39.19
39.02
21.03
20.71
O
NH
Me
Me
5ag
^{13}C NMR, 125 MHz, DMSO-D$_6$
f1 (ppm)
f1 (ppm)


12.04
8.32
8.31
8.31
7.72
7.72
7.71
7.69
7.69
7.61
7.59
7.58
7.57
7.55
7.51
7.50
7.49
7.49
7.47
7.47
7.46
7.40
7.38
7.36
7.35
7.32
7.32
7.30
7.27
7.25
7.24
7.23
7.22
7.15
3.28
2.50
O
NH
F
F
5ah
1H NMR, 500 MHz, DMSO-D6
1.03
1.00
1.02
2.08
2.05
2.03
1.03
2.04
1.01
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 0.0
f1 (ppm)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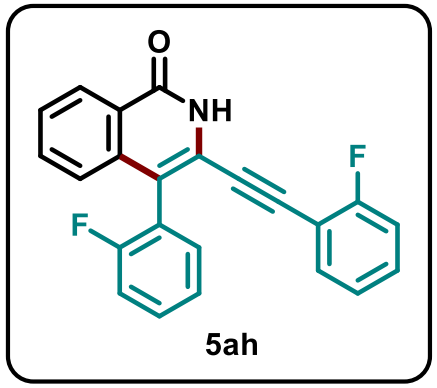
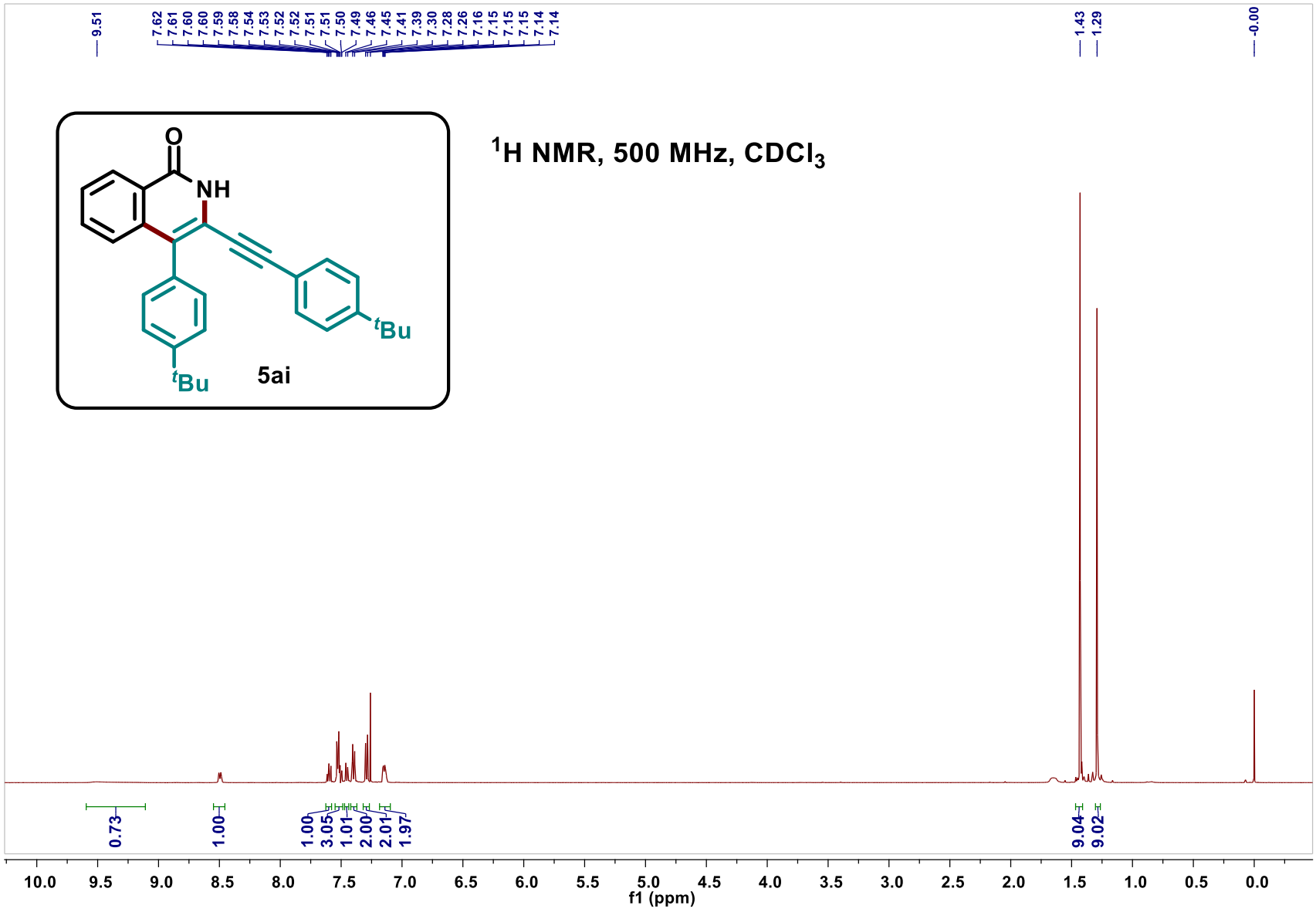
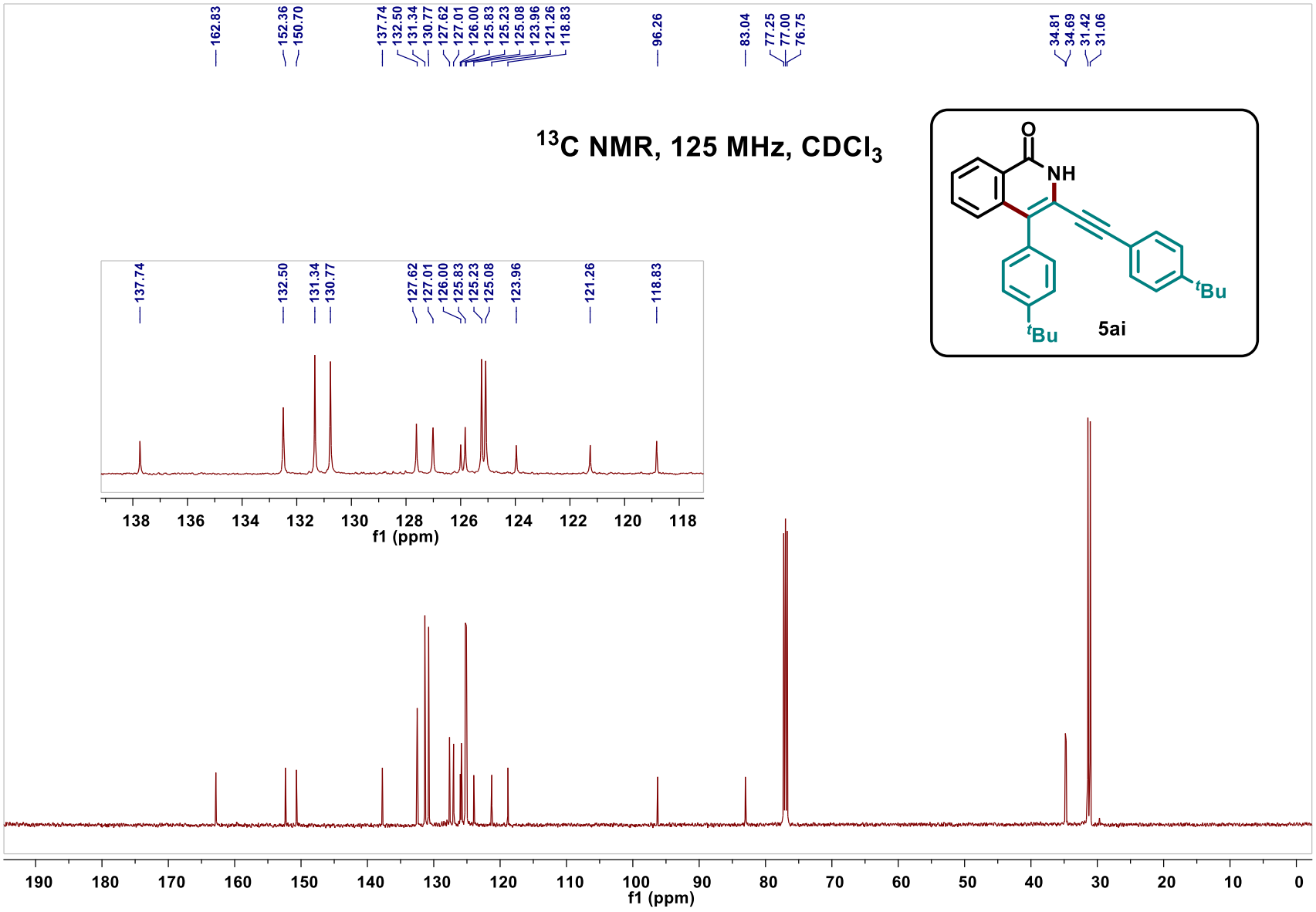
161.10
160.52
159.07
136.28
133.25
133.15
132.91
131.90
131.83
130.72
130.66
127.63
127.06
126.08
124.84
124.75
124.55
122.25
121.84
116.65
115.74
109.40
109.27
88.72
87.40
87.38
40.02
39.85
39.69
39.52
39.35
39.19
39.02
^{13}C NMR, 125 MHz, DMSO-D$_6$
O
NH
F
F
5ah
f1 (ppm)
f1 (ppm)

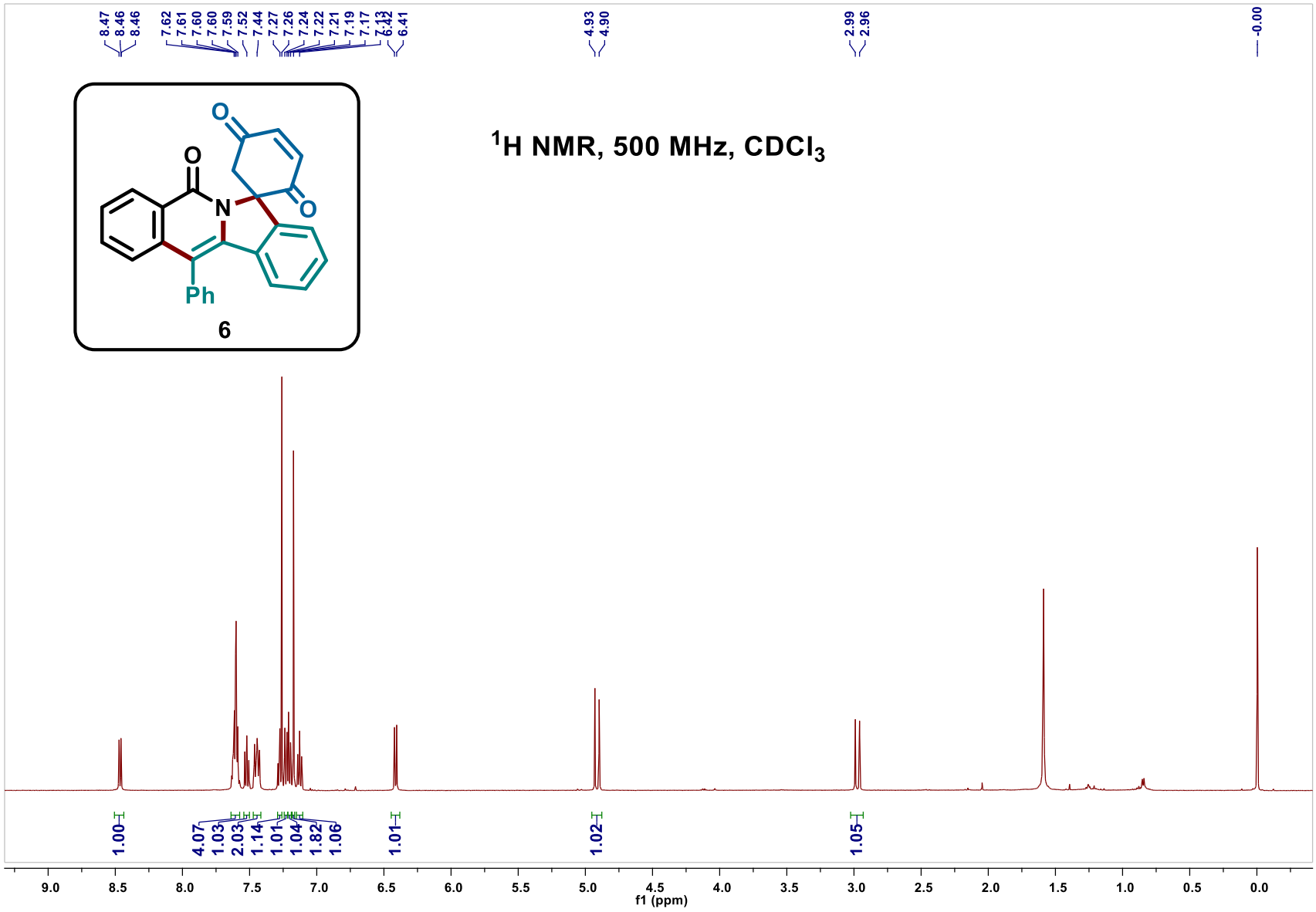


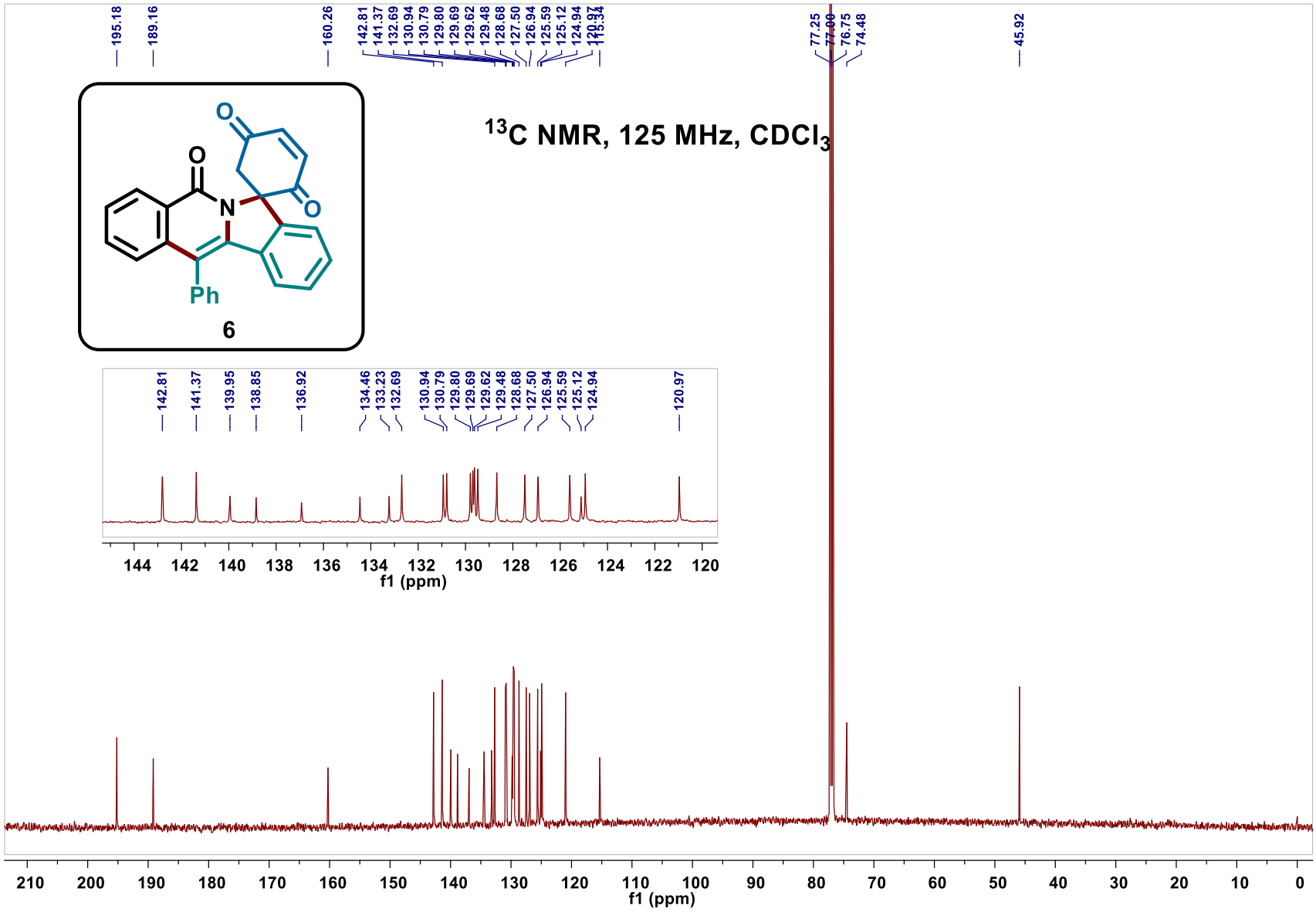
5ah

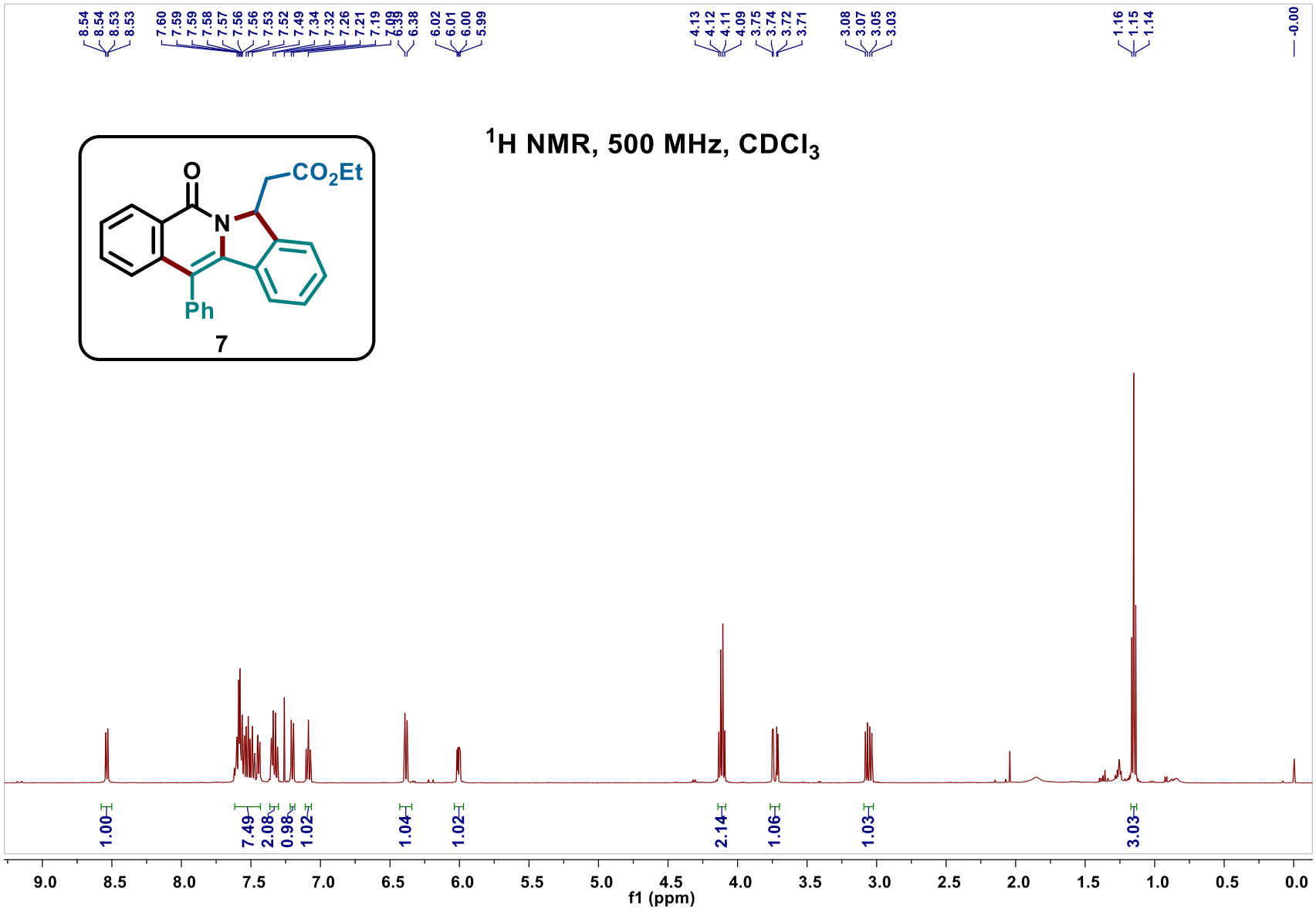
^{19}F NMR, 470 MHz, DMSO-D$_6$

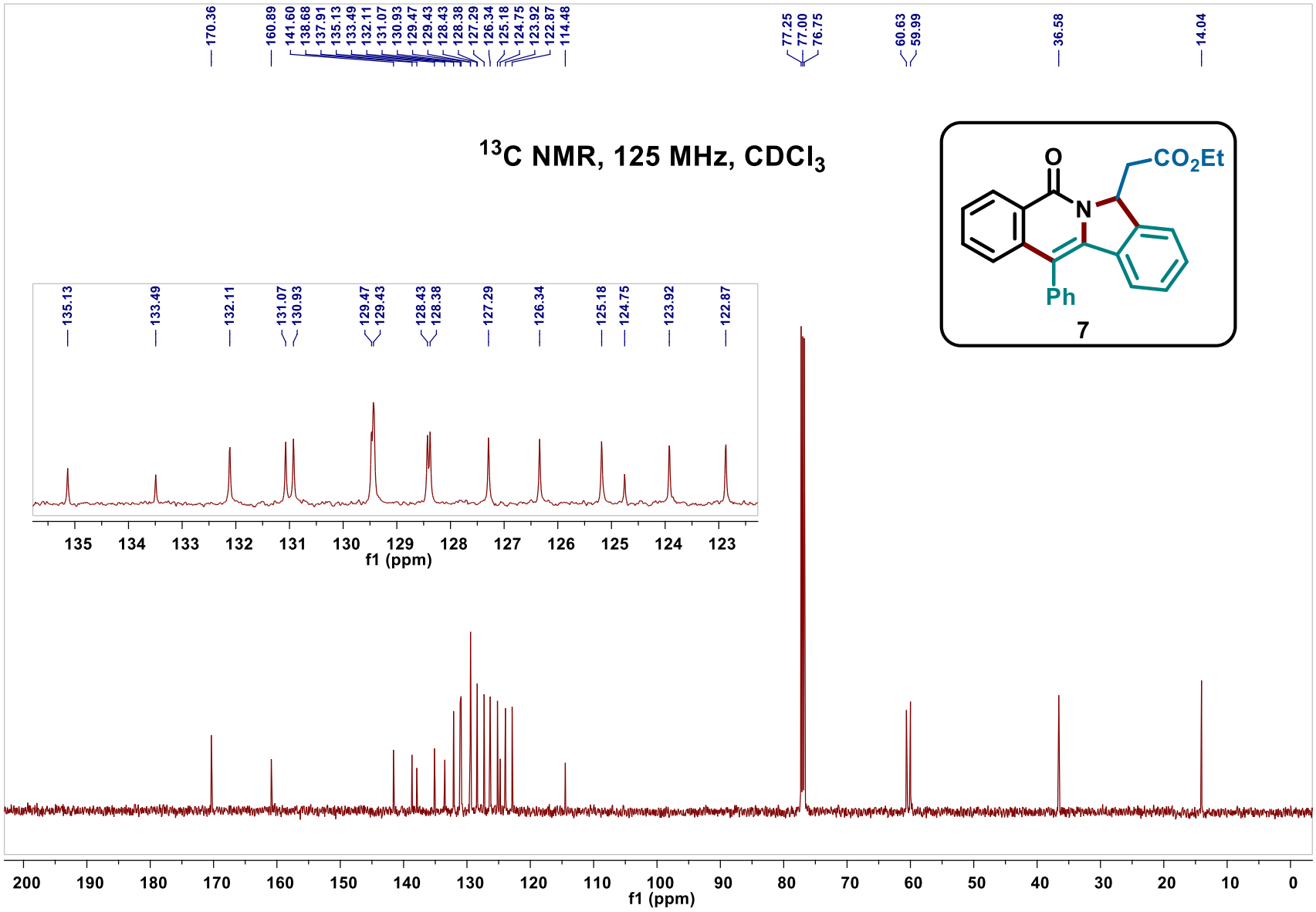

9.51
7.62
7.61
7.60
7.60
7.59
7.58
7.54
7.53
7.52
7.52
7.51
7.51
7.50
7.49
7.46
7.45
7.41
7.39
7.30
7.28
7.26
7.16
7.15
7.15
7.15
7.14
7.14
1.43
1.29
-0.00
O
NH
tBu
tBu
5ai
1H NMR, 500 MHz, CDCl3
0.73
1.00
1.00
3.05
1.01
2.00
2.01
1.97
9.04
9.02
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

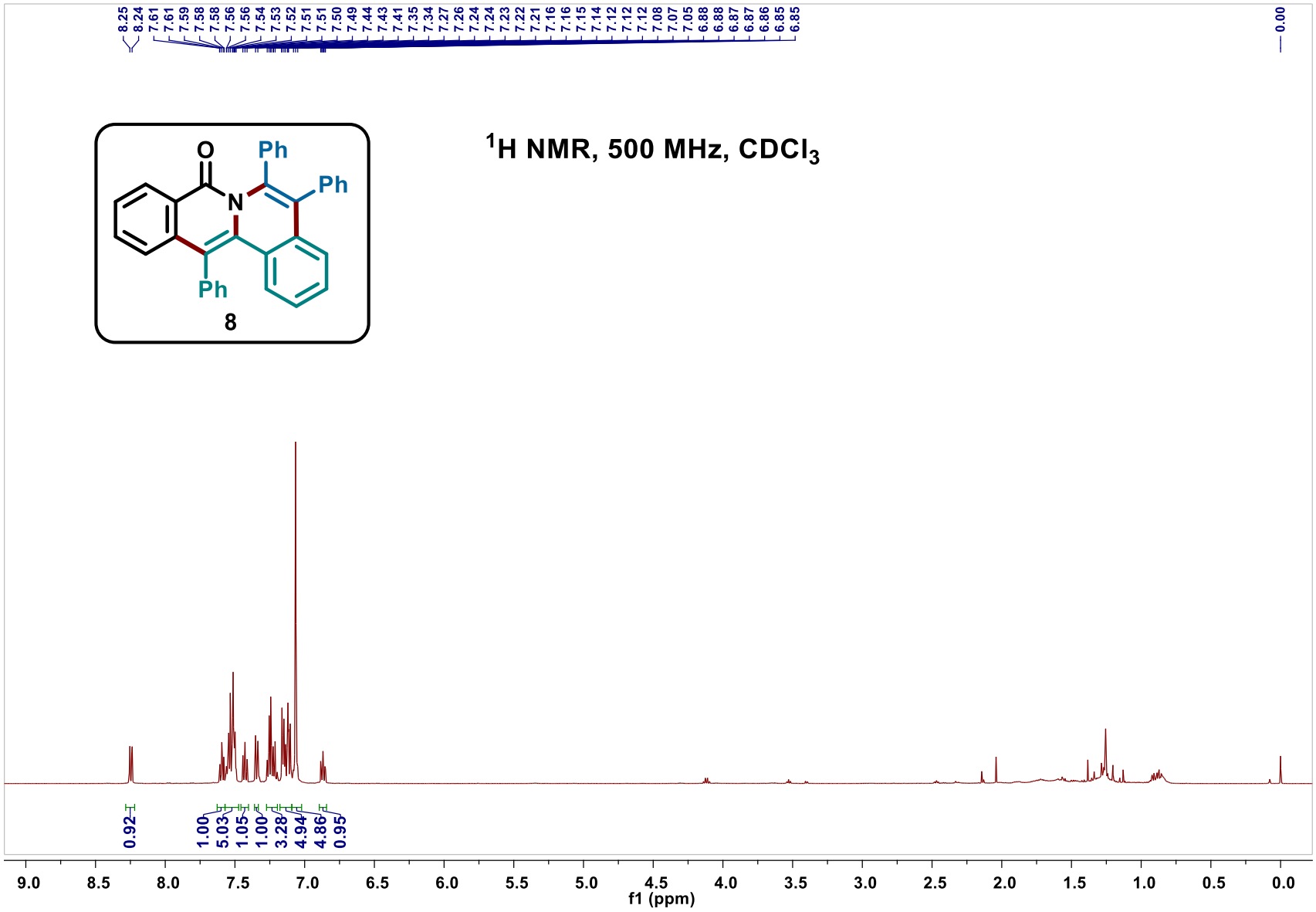

162.83
152.36
150.70
137.74
132.50
131.34
130.77
127.62
127.01
126.00
125.83
125.23
125.08
123.96
121.26
118.83
96.26
83.04
77.25
77.00
76.75
34.81
34.69
31.42
31.06
13C NMR, 125 MHz, CDCl3
O
NH
tBu
tBu
5ai
f1 (ppm)
f1 (ppm)

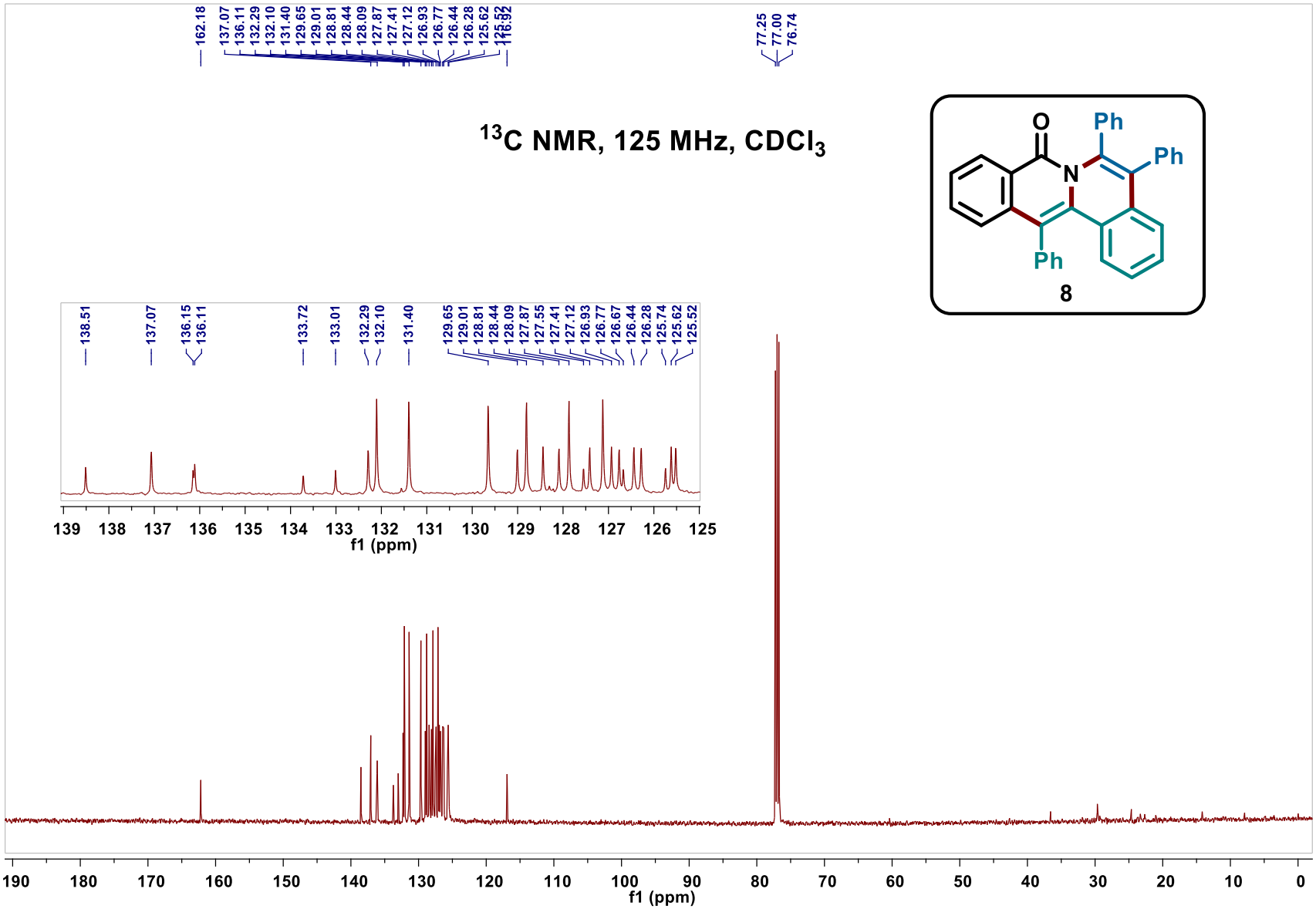

8.47
8.46
8.46
7.62
7.61
7.60
7.60
7.59
7.52
7.44
7.27
7.26
7.24
7.22
7.21
7.19
7.17
7.13
7.12
6.41
4.93
4.90
2.99
2.96
-0.00
O
O
N
O
Ph
6
^{1}H NMR, 500 MHz, CDCl$_3$
1.00
4.07
1.03
2.03
1.14
1.01
1.04
1.82
1.06
1.01
1.02
1.05
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

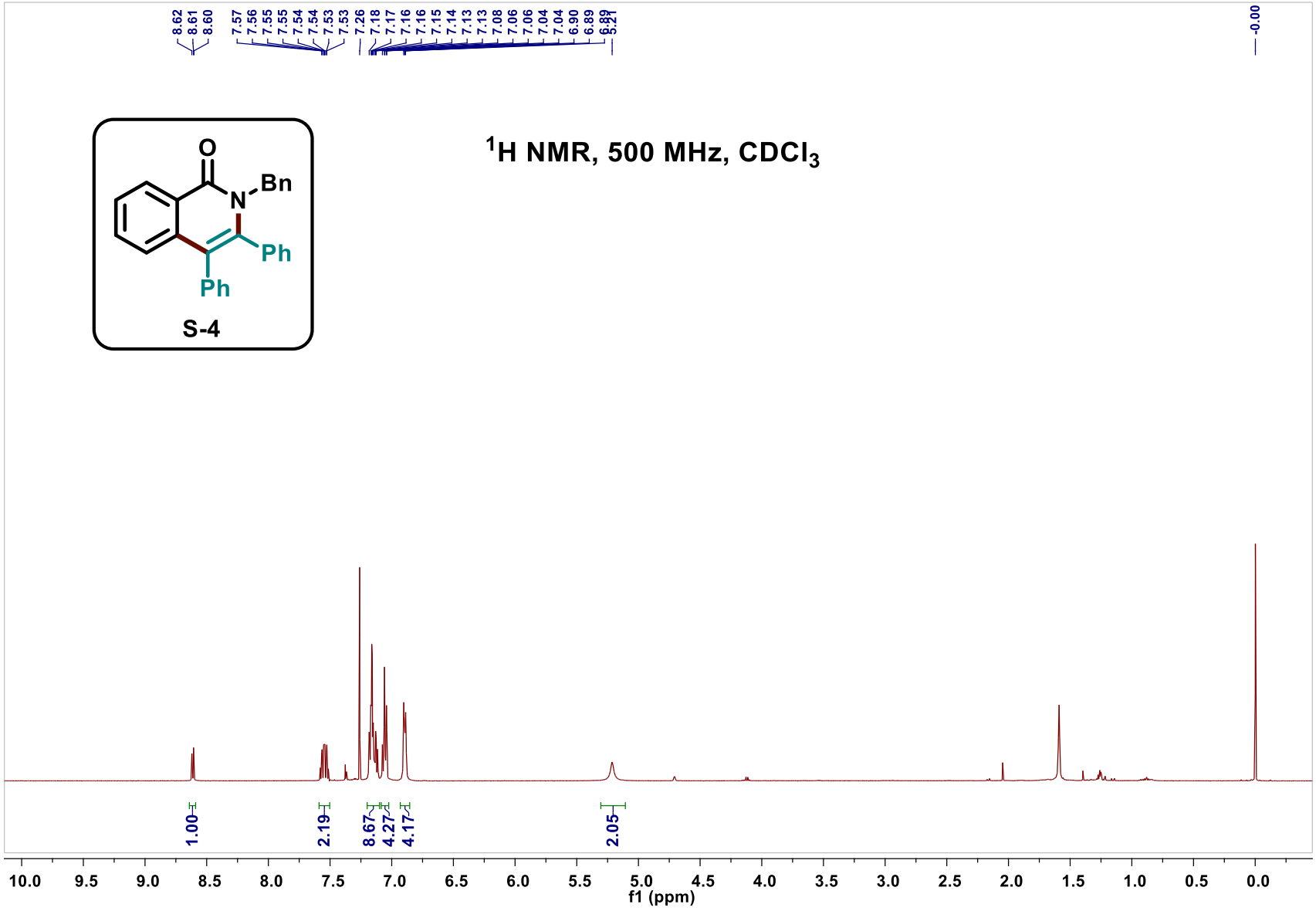

195.18
189.16
160.26
142.81
141.37
132.69
130.94
130.79
129.80
129.69
129.62
129.48
128.68
127.50
126.94
125.59
125.12
124.94
120.97
115.34
77.25
77.00
76.75
74.48
45.92
13C NMR, 125 MHz, CDCl3
O
O
N
O
Ph
6
142.81
141.37
139.95
138.85
136.92
134.46
133.23
132.69
130.94
130.79
129.80
129.69
129.62
129.48
128.68
127.50
126.94
125.59
125.12
124.94
120.97
144 142 140 138 136 134 132 130 128 126 124 122 120
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

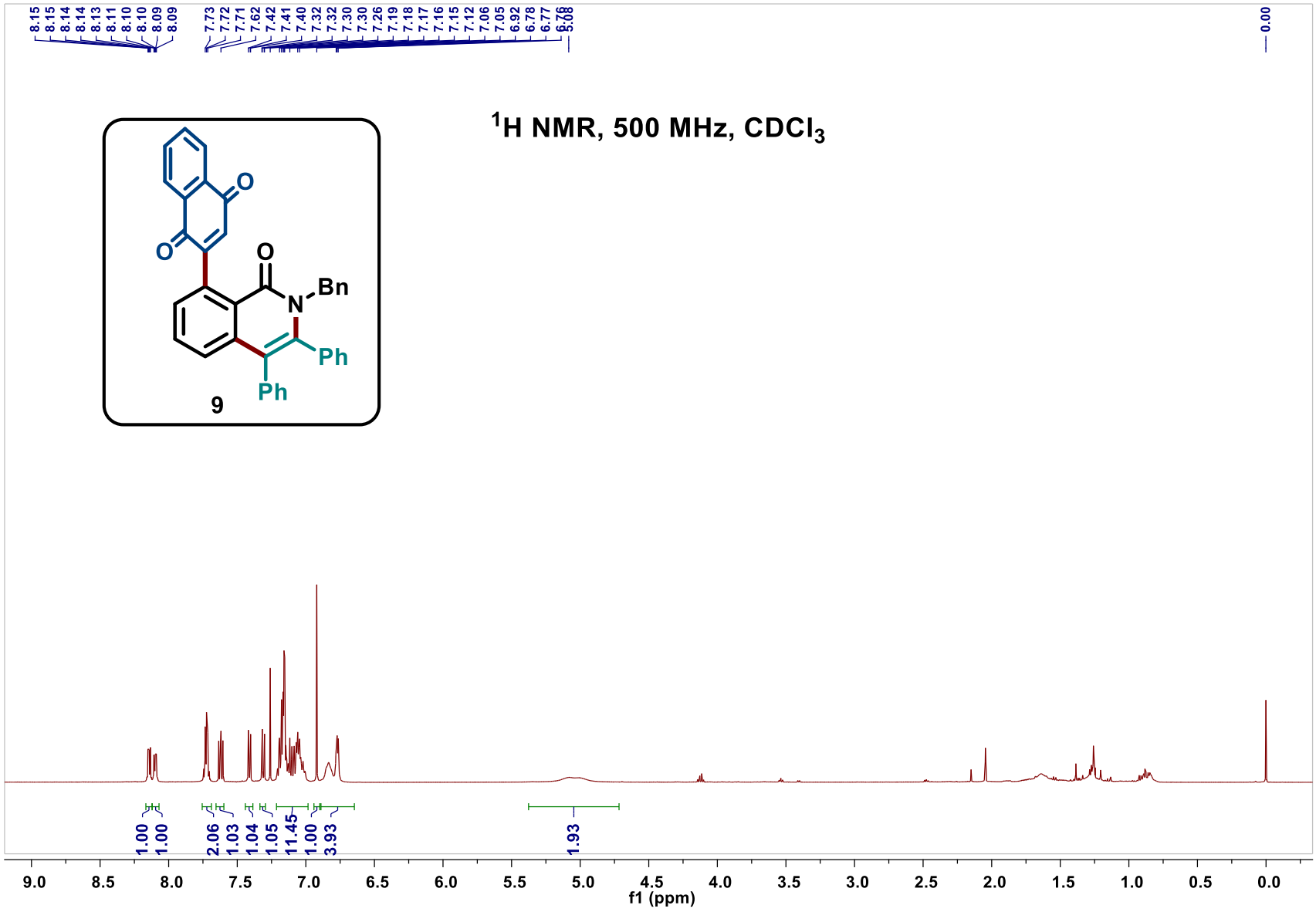

8.54 8.54 8.53 8.53
7.60 7.59 7.59 7.58 7.57 7.56 7.56 7.53 7.52 7.49 7.34 7.32 7.26 7.21 7.19 7.09 6.38
6.02 6.01 6.00 5.99
4.13 4.12 4.11 4.09
3.75 3.74 3.72 3.71
3.08 3.07 3.05 3.03
1.16 1.15 1.14
-0.00
O
N
CO2Et
Ph
7
1H NMR, 500 MHz, CDCl3
1.00 7.49 2.08 0.98 1.02 1.04 1.02 2.14 1.06 1.03 3.03
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

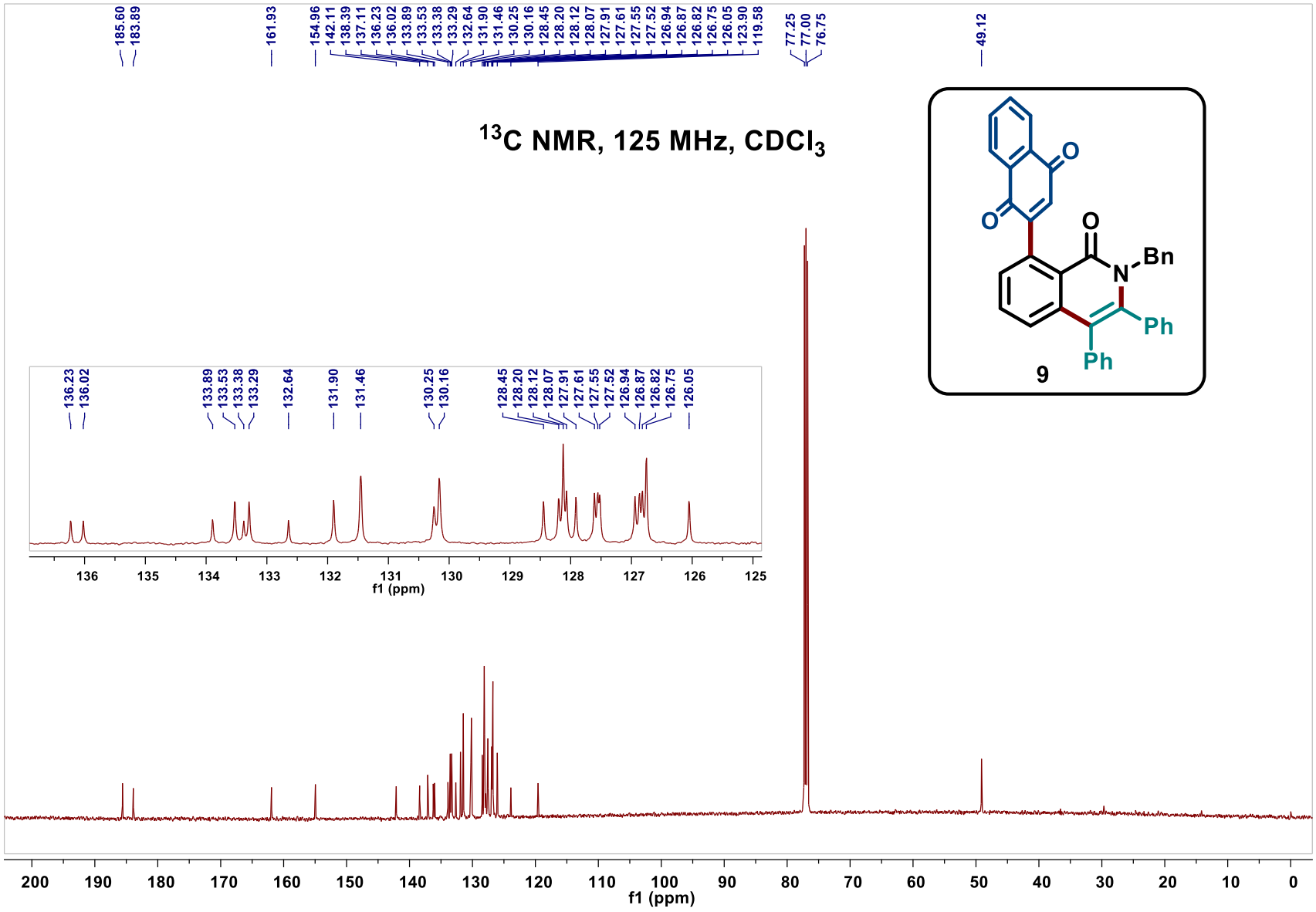

170.36
160.89
141.60
138.68
137.91
135.13
133.49
132.11
131.07
130.93
129.47
129.43
128.43
128.38
127.29
126.34
125.18
124.75
123.92
122.87
114.48
77.25
77.00
76.75
60.63
59.99
36.58
14.04
13C NMR, 125 MHz, CDCl3
O
CO2Et
N
Ph
7
f1 (ppm)
f1 (ppm)


8.25
8.24
7.61
7.61
7.59
7.58
7.58
7.56
7.56
7.54
7.53
7.52
7.51
7.51
7.50
7.49
7.44
7.43
7.41
7.35
7.34
7.27
7.26
7.24
7.24
7.23
7.22
7.21
7.16
7.16
7.15
7.14
7.12
7.12
7.12
7.08
7.07
7.05
6.88
6.88
6.87
6.87
6.86
6.85
6.85
0.00
O
Ph
Ph
N
Ph
8
1H NMR, 500 MHz, CDCl3
0.92
1.00
5.03
1.05
1.00
3.28
4.94
4.86
0.95
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


162.18
137.07
136.11
132.29
132.10
131.40
129.65
129.01
128.81
128.44
128.09
127.87
127.41
127.12
126.93
126.77
126.44
126.28
125.62
125.52
77.25
77.00
76.74
13C NMR, 125 MHz, CDCl3
O
Ph
Ph
N
Ph
8
138.51
137.07
136.15
136.11
133.72
133.01
132.29
132.10
131.40
129.65
129.01
128.81
128.44
128.09
127.87
127.55
127.41
127.12
126.93
126.77
126.67
126.44
126.28
125.74
125.62
125.52
139 138 137 136 135 134 133 132 131 130 129 128 127 126 125
f1 (ppm)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


8.62
8.61
8.60
7.57
7.56
7.55
7.55
7.54
7.54
7.53
7.53
7.26
7.18
7.17
7.16
7.16
7.15
7.14
7.13
7.13
7.08
7.06
7.06
7.04
7.04
6.90
6.89
6.89
5.21
0.00
O
N
Bn
Ph
Ph
S-4
1H NMR, 500 MHz, CDCl3
1.00
2.19
8.67
4.27
4.17
2.05
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
0.0
f1 (ppm)


8.15 8.15 8.14 8.14 8.13 8.11 8.10 8.10 8.09 8.09
7.73 7.72 7.71 7.62 7.42 7.41 7.40 7.32 7.32 7.30 7.30 7.26 7.19 7.18 7.17 7.16 7.15 7.12 7.06 7.05 6.92 6.78 6.77
0.00
O
O
O
N
Bn
Ph
Ph
9
1H NMR, 500 MHz, CDCl3
1.00 1.00 2.06 1.03 1.04 1.05 11.45 1.00 3.93 1.93
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


185.60
183.89
161.93
154.96
142.11
138.39
137.11
136.23
136.02
133.89
133.53
133.38
133.29
132.64
131.90
131.46
130.25
130.16
128.45
128.20
128.12
128.07
127.91
127.61
127.55
127.52
126.94
126.87
126.82
126.75
126.05
123.90
119.58
77.25
77.00
76.75
49.12
13C NMR, 125 MHz, CDCl3
O
O
O
N
Bn
Ph
Ph
9
f1 (ppm)
f1 (ppm)