Electronic Supplementary Material (ESI) for ChemComm.

Cu-catalyzed C(sp^2)-N-bond coupling of boronic acids and cyclic imides

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Supplementary information

Contents

General considerations

All reactions were performed with open access to air unless otherwise stated. Commercially available reagents and solvents were purchased from Sigma-Aldrich, TCI, Alfa Aesar, AK Scientific and Fluorochem were used without further purification unless stated otherwise. Hexanes were distilled prior to use. Thin layer chromatography was performed on 60 F_{254} silica coated aluminum plates from Merck and visualized using UV-light or $KMnO_4$-stain (1.5 g $KMnO_4$, 10 g K_2CO_3 and 1.25 mL 10% NaOH in 200 mL water). Flash chromatography was performed on silica gel from Merck (Silicagel 60, 40-63 µm) using fritted glass columns.

^{1}H and ^{13}C NMR spectra were recorded using Bruker AVI600 and AVII600 spectrometers.^{19}F NMR spectra were recorded using a Bruker AVIIIHD400 spectrometer. Spectra were calibrated using the residual solvent peaks for $CDCl_3$ (^{1}H: 7.26 ppm; ^{13}C: 77.00 ppm), DMSO-d_6 (^{1}H: 2.50 ppm; ^{13}C: 39.52 ppm), CD_3CN (^{1}H: 1.94 ppm; ^{13}C: 1.32/118.26 ppm), acetone-d_6 (^{1}H: 2.05 ppm; ^{13}C: 29.84/206.26 ppm) and CD_3OD (^{1}H: 3.31 ppm; ^{13}C: 49.00 ppm). ^{19}F NMR spectra were unreferenced. All spectra were recorded at 298 K. Chemical shifts are reported in parts per million (ppm, δ). ^{1}H NMR multiplicities are reported as singlet (s), broad singlet (br s), doublet (d), triplet (t), quartet (q), pentet (p), sextet (sext), heptet (h), doublet of doublets (dd), triplet of doublets (td), quartet of doublets (qd), doublet of doublet of doublets (ddd), doublet of triplet of doublets (dtd), AB quartet (ABq), multiplet (m). Coupling constants (*J*) are reported in hertz.

^{1}H-^{15}N HMBC NMR was used to determine the *N*-selectivity in compounds possessing several *N*- or *O*-nucleophilic sites. A few selected examples are included. The ^{1}H-^{15}N HMBC NMR spectra were recorded using Bruker AVI600 and AVII600 spectrometers and referenced externally to nitromethane (0 ppm). The spectra were acquired using Bruker hmbcf3gplpndqf pulse program.

FTIR spectra were recorded in ATR (Bruker ATR A225/Q) on a Vertex 80 Bruker infrared spectrophotometer, equipped with a DTGS detector; 32 interferograms (recorded at 4 cm^{-1} resolution) were typically averaged for each spectrum.

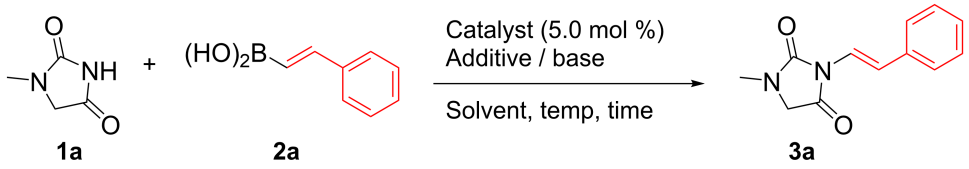
High-resolution mass spectra (HRMS) were obtained by electron spray ionization (ESI) on Bruker Daltonik GmbH MAXIS II ETD spectrometer.

Optical rotations were measured on a polarimeter using a 10 cm cell with a Na 589 nm filter. The specific solvents and concentrations (in g/100 mL) are indicated.

Melting point for crystalline compounds were measured on a Stuart SMP10 melting point apparatus and are reported uncorrected.

Single crystal diffraction data were acquired on a Bruker D8 Venture equipped with a Photon 100 detector by using Mo K_α radiation (λ = 0.71073 Å) from an Incoatec iµS microsource. Data reduction was performed with the Bruker Apex3 Suite. Structure **3m**, **3j'**, **3q** and **4h** were solved with ShelXT and refined with ShelXL. Structure **7a** was solved with ShelXT and refined with Olex2. Olex2 was used as user interface. Molecular graphics were produced with Diamond v 4.6.4.

Key controls and optimization data for the general procedure



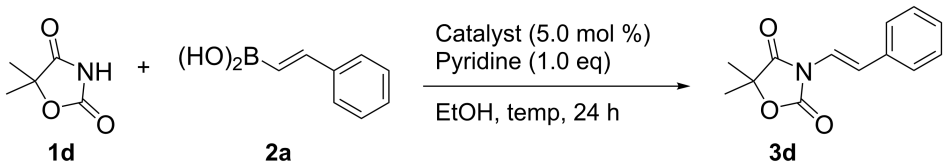
1a **2a** **3a**

#	Catalyst	**2a** (eq.)	Additive/ base (eq.)	Solvent	Temp (°C)	Time (h)	Yield (%)[a]
1	$Cu(OTf)_2$	3.0	Py (1.0)	EtOH	25	9/15	98/97[b]
2	$Cu(OTf)_2$	2.0	Py (1.0)	EtOH	25	24	94
3	$Cu(OTf)_2$	1.5	Py (1.0)	EtOH	40	24	45
4	$Cu(OTf)_2$	1.2	Py (1.0)	EtOH	40	24	42
5	$Cu(OTf)_2$	1.0	Py (1.0)	EtOH	25	24	41
6	$Cu(OTf)_2$	1.0	Py (1.0)	EtOH	40	24	71
7	$Cu(OTf)_2$	0.5	Py (1.0)	EtOH	40	24	28
8	CuI	3.0	Py (1.0)	EtOH	25	24	0
9	CuCl	3.0	Py (1.0)	EtOH	25	24	95
10	$CuCl_2$	3.0	Py (1.0)	EtOH	25	24	77
11	$Cu(NO_3)_2$[c]	3.0	Py (1.0)	EtOH	25	24	96
12	$Cu(OTf)_2$	3.0	Py (0.2)	EtOH	25	18	47
13	$Cu(OTf)_2$	3.0	Et_3N (1.0)	EtOH	25	24	8
14	$Cu(OTf)_2$	3.0	DIPEA (1.0)	EtOH	25	16	1
15	$Cu(OTf)_2$	3.0	chxn[d] (1.0)	EtOH	25	16	0
16	$Cu(OTf)_2$	3.0	DTBP[e] (1.0)	EtOH	25	18	8
17	$Cu(OTf)_2$	3.0	K_2CO_3 (1.0)	EtOH	25	18	25
18	$Cu(OTf)_2$	3.0	K_2CO_3 (1.0)	EtOH	40	18	52
19	$Cu(OTf)_2$	3.0	*t*-BuOK (1.0)	EtOH	25	18	22
20	-	3.0	*t*-BuOK (2.0)	EtOH	25	24	0
21	$Cu(OTf)_2$	3.0	LiHMDS (1.0)	EtOH	25	18	3
22	$Cu(OTf)_2$	3.0	LiHMDS (1.0)	THF	25	24	0
23	$Cu(OTf)_2$	3.0	Py (1.0)	DMF	25	24	26
24	$Cu(OTf)_2$	3.0	Py (1.0)	PhMe	25	24	0
25	$Cu(OTf)_2$	3.0	Py (1.0)	*i*-PrOH	25	24	83
26	$Cu(OTf)_2$	3.0	-	EtOH	40	23	8
27	-	3.0	Py (1.0)	EtOH	40	24	0
28	-	3.0	Py (1.0)	EtOH	80	24	0
29	-	3.0	-	EtOH	40	23	0

Green entry: Optimized conditions. Blue entries: Entries included in Table 1 in the manuscript.

Conditions: *N*-Methylhydantoin **1a** (0.20 mmol, 1.0 equiv.), boronic acid **2a** (as specified), catalyst (0.010 mmol, 0.050 equiv.), additive/base (as specified) in solvent (as specified, 1 mL). [a] ^{1}H NMR yield using mesitylene as internal standard, [b] Isolated yield. [c] The (hemi)pentahydrate salt was used. [d] chxn = *trans*-1,2-Diaminocyclohexane. [e] DTBP = 2,6-Di-*tert*-butylpyridine.

Synthesis of 3d under various conditions



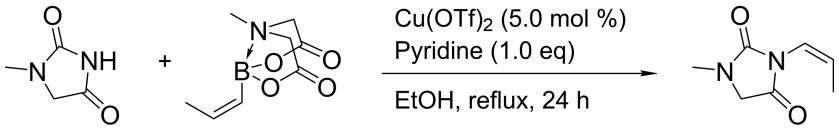
1d **2a** **3d**

#	Catalyst	**2a** (eq.)	Temp (°C)	Yield (%)[a]
1	$Cu(OTf)_2$	1.0	40	37
2	CuCl	3.0	25	80

Conditions: 2,4-Oxazolidinedione **1d** (0.20 mmol, 1.0 equiv.), boronic acid **2a** (as specified), catalyst (0.010 mmol, 0.050 equiv.), pyridine (0.20 mmol, 1.0 equiv) in ethanol (1 mL). [a] ^{1}H NMR yield using mesitylene as internal standard.

C(sp^2)-N couplings with other boron containing reagents

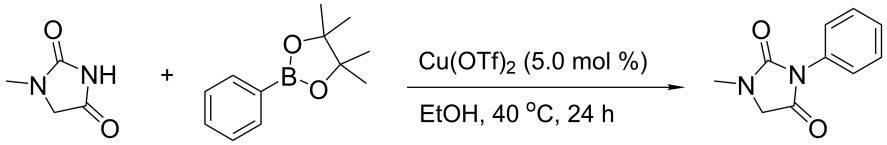
N-alkenylation with MIDA boronate ester failed to convert hydantoin **1a** to the desired alkenylated product (Scheme SI-1).



1a MIDA boronate ester (3.0 eq) 0 %

Scheme SI-1: *N*-alkenylation with MIDA boronate ester. Conditions: *N*-Methylhydantoin **1a** (0.20 mmol, 1.0 equiv.), MIDA boronate ester (0.6 mmol, 3.0 equiv.), $Cu(OTf)_2$ (0.010 mmol, 0.050 equiv.), pyridine (0.20 mmol, 1.0 equiv) in ethanol (1 mL).

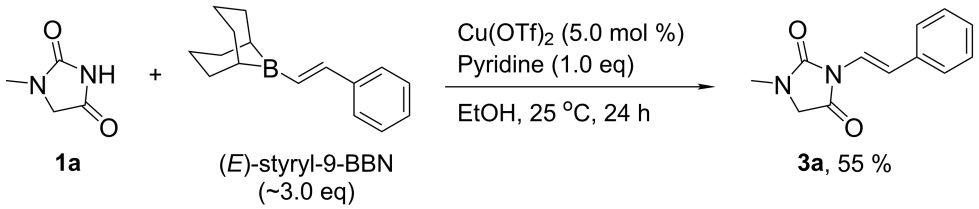
12 % of hydantoin **7g** was isolated in the arylation of hydantoin **1a** with phenyl pinacol boronate ester (scheme SI-2).



1a Pinacol boronate ester (1.1 eq) **7g**, 12 %

Scheme SI-2: *N*-arylation with pinacol boronate ester. Conditions: *N*-Methylhydantoin **1a** (0.20 mmol, 1.0 equiv.), pinacol boronate ester (0.22 mmol, 1.1 equiv.), $Cu(OTf)_2$ (0.010 mmol, 0.050 equiv.) in ethanol (1 mL).

55 % of hydantoin **3a** was isolated in the alkenylation of hydantoin **1a** using (*E*)-styryl-9-BBN as alkenylating reagent (scheme SI-3).



1a (*E*)-styryl-9-BBN (~3.0 eq) **3a**, 55 %

Scheme SI-3: *N*-alkenylation with (*E*)-styryl-9-BBN. Conditions: *N*-Methylhydantoin **1a** (0.20 mmol, 1.0 equiv.), ~0.5 M THF-solution of (*E*)-styryl-9-BBN (~0.60 mmol, ~3.0 equiv.), $Cu(OTf)_2$ (0.010 mmol, 0.050 equiv.) in ethanol (1 mL). (*E*)-styryl-9-BBN was prepared according to literature[1].

Single crystal X-ray diffraction data for 3m (CCDC 2099681)

In a glass vial, **3m** was dissolved in deuterated chloroform. The vial was left open and single crystals were grown by slow evaporation of the solvent in room temperature over night.

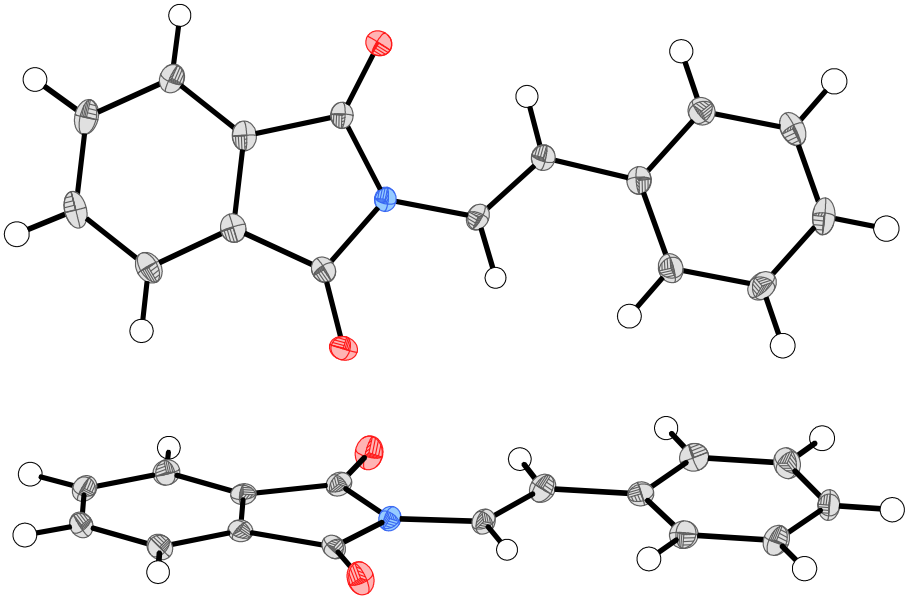


Figure SI-1: ORTEP representation of **3m**. Ellipsoids are of 50% probability.

Table SI-1: Crystal data and structure refinement for **3m**

Crystal data

Empirical formula	$C_{32}H_{22}N_2O_4$
Formula weight	498.51
Crystal color, shape	Yellow, plate
Crystal size (mm^3)	0.3 × 0.2 × 0.16
Crystal system, space group	Orthorhombic, $Pca2_1$
a, b, c (Å)	28.5959(19), 7.4128(5), 11.4628(8)
α, β, γ (°)	90, 90, 90
Volume ($Å^3$)	2429.8(3)
Z	4
Temperature (K)	104.6
$\rho_{calc}g$ (cm^3)	1.363
μ (mm^{-1})	0.091
F(000)	1040.0
Radiation	MoKα (λ = 0.71073)

Data collection and refinement

2Θ range for data collection (°)	4.554 to 52.738
Index ranges	-35 ≤ h ≤ 35, -9 ≤ k ≤ 8, -14 ≤ l ≤ 9
Reflections collected	16187
Independent reflections	4117 (R_{int} = 0.0222, R_{sigma} = 0.0230)
Data/restraints/parameters	4117/1/343
Goodness-of-fit on F^2	0.867
Final R indexes (I>=2σ (I))	R_1 = 0.0320, wR_2 = 0.0937
Final R indexes (all data)	R_1 = 0.0348, wR_2 = 0.0969
Largest diff. peak/hole ($e/Å^{-3}$)	0.24/-0.19
Flack parameter	0.3(3)

Single crystal X-ray diffraction data for 3j' (CCDC 2099684)

In a glass vial, **3j'** was dissolved in deuterated chloroform. The vial was left open and single crystals were grown by slow evaporation of the solvent in room temperature over night.

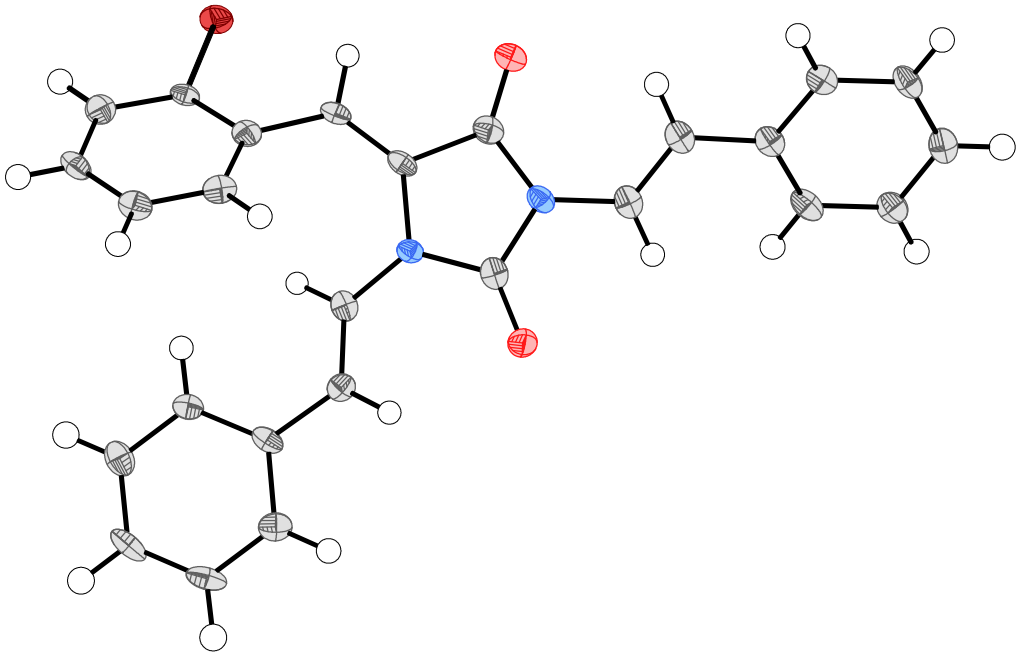


Figure SI-3: ORTEP representation of **3j'**. Ellipsoids are of 50% probability.

Table SI-3: Crystal data and structure refinement for **3j'**

Crystal data

Empirical formula	$C_{26}H_{19}BrN_2O_2$
Formula weight	471.34
Crystal color, shape	Colorless, plate
Crystal size (mm^3)	1.1 × 0.43 × 0.08
Crystal system, space group	Triclinic, P-1
a, b, c (Å)	9.2065(11), 10.3700(13), 12.0590(15)
α, β, γ (°)	77.804(3), 72.890(3), 73.581(3)
Volume ($Å^3$)	1045.1(2)
Z	2
Temperature (K)	100.0
ρ_{calc}g (cm^3)	1.498
μ (mm^{-1})	1.993
F(000)	480.0
Radiation	MoKα (λ = 0.71073)

Data collection and refinement

2Θ range for data collection (°)	4.14 to 50.27
Index ranges	$-10 \le h \le 10$, $-11 \le k \le 12$, $0 \le l \le 14$
Reflections collected	4754
Independent reflections	3678
Data/restraints/parameters	3678/0/281
Goodness-of-fit on F^2	1.029
Final R indexes (I>=2σ (I))	R_1 = 0.0531, wR_2 = 0.1253
Final R indexes (all data)	R_1 = 0.0765, wR_2 = 0.1383
Largest diff. peak/hole ($e/Å^{-3}$)	0.81/-0.63

Single crystal X-ray diffraction data for 3r (CCDC 2099683)

In a glass vial, **3r** was dissolved in deuterated chloroform. The vial was left open and single crystals were grown by slow evaporation of the solvent in room temperature over night.

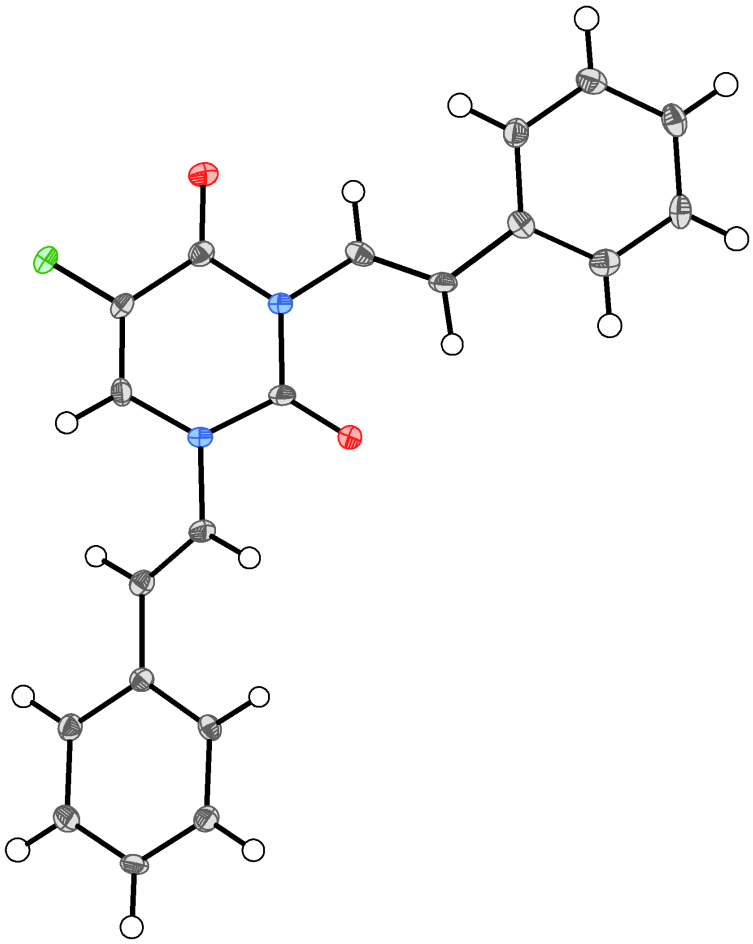


Figure SI-4: ORTEP representation of **3r**. Ellipsoids are of 50% probability.

Table SI-4: Crystal data and structure refinement for **3r**

Crystal data

Empirical formula	$C_{20}H_{15}FN_2O_2$
Formula weight	334.34
Crystal color, shape	Colorless, needle
Crystal size (mm^3)	0.44 × 0.19 × 0.18
Crystal system, space group	Trigonal, R3c
a, b, c (Å)	33.8915(10), 33.8915(10), 7.0535(13)
α, β, γ (°)	90, 90, 120
Volume ($Å^3$)	7016.4(14)
Z	18
Temperature (K)	99.9
$\rho_{calc}g$ (cm^3)	1.424
μ (mm^{-1})	0.101
F(000)	3132.0
Radiation	MoKα (λ = 0.71073)

Data collection and refinement

2Θ range for data collection (°)	4.164 to 52.774
Index ranges	-41 ≤ h ≤ 0, -35 ≤ k ≤ 21, -8 ≤ l ≤ 8
Reflections collected	5543
Independent reflections	5543 (R_{sigma} = 0.0633)
Data/restraints/parameters	5543/1/228
Goodness-of-fit on F^2	1.052
Final R indexes (I>=2σ (I))	R_1 = 0.0537, wR_2 = 0.0966
Final R indexes (all data)	R_1 = 0.0803, wR_2 = 0.1076
Largest diff. peak/hole ($e/Å^{-3}$)	0.25/-0.27
Flack parameter	-0.2(6)

Single crystal X-ray diffraction data for 4h (CCDC 2099682)

In a glass vial, **4h** was dissolved in deuterated chloroform. The vial was left open and single crystals were grown by slow evaporation of the solvent in room temperature over night.

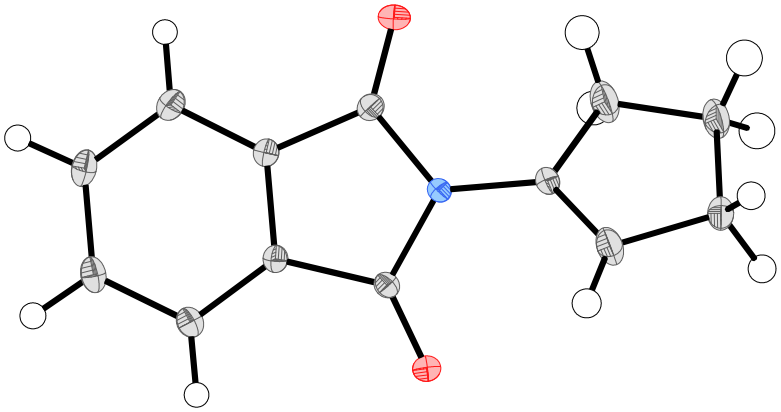


Figure SI-2: ORTEP representation of **4h**. Ellipsoids are of 50% probability.

Table SI-2: Crystal data and structure refinement for **4h**

Crystal data

Empirical formula	$C_{13}H_{11}NO_2$
Formula weight	213.23
Crystal color, shape	Colorless, plate
Crystal size (mm^3)	0.3 × 0.2 × 0.16
Crystal system, space group	Monoclinic, $P2_1/c$
a, b, c (Å)	11.9019(13), 11.4470(13), 7.4837(8)
α, β, γ (°)	90, 101.078(3), 90
Volume ($Å^3$)	1000.59(19)
Z	4
Temperature (K)	103.7
$\rho_{calc}g$ (cm^3)	1.415
μ (mm^{-1})	0.096
F(000)	448.0
Radiation	MoKα (λ = 0.71073)

Data collection and refinement

2Θ range for data collection (°)	4.982 to 61.008
Index ranges	-17 ≤ h ≤ 16, -16 ≤ k ≤ 16, -10 ≤ l ≤ 10
Reflections collected	20966
Independent reflections	3055 (R_{int} = 0.0354, R_{sigma} = 0.0217)
Data/restraints/parameters	3055/0/145
Goodness-of-fit on F^2	1.046
Final R indexes (I>=2σ (I))	R_1 = 0.0461, wR_2 = 0.1215
Final R indexes (all data)	R_1 = 0.0549, wR_2 = 0.1272
Largest diff. peak/hole ($e/Å^{-3}$)	0.42/-0.50

Single crystal X-ray diffraction data for 7a (CCDC 2099686)

In small a glass vial, **7a** was dissolved in dichloromethane. The small, open vial was placed inside a larger vial containing hexane. The larger vial was sealed and single crystals of **7a** were grown by slow diffusion of the hexane into the saturated solution of **7a**.

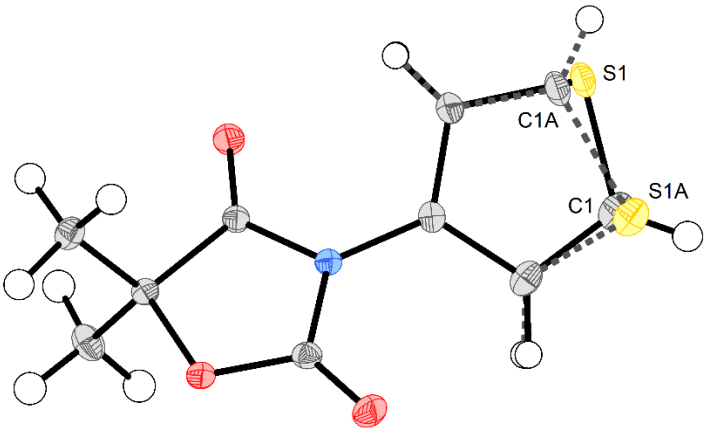


Figure SI-5: ORTEP representation of **7a**. Ellipsoids are of 50% probability. Structural disorder due to rotation of the thiophene ring is shown. The site occupancy factors for the two positions were refined to 0.856 (including atoms C1 and S1) and 0.144 (including atoms C1A and S1A). The minor conformer is displayed with grey dashed bonds.

Table SI-5: Crystal data and structure refinement for **7a**

Crystal data

Empirical formula	$C_9H_9NO_3S$
Formula weight	211.24
Crystal color, shape	Colorless, needle
Crystal size (mm^3)	0.44 × 0.19 × 0.18
Crystal system, space group	Orthorhombic, Pbca
a, b, c (Å)	13.5471(10), 7.5236(5), 18.4113(14)
α, β, γ (°)	90, 90, 90
Volume ($Å^3$)	1876.5(2)
Z	8
Temperature (K)	100.0
$\rho_{calc}g$ (cm^3)	1.4953
μ (mm^{-1})	0.323
F(000)	881.5
Radiation	MoKα (λ = 0.71073)

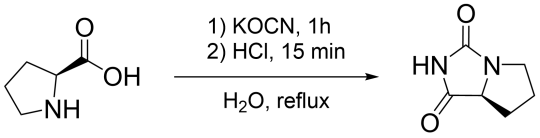
Data collection and refinement

2Θ range for data collection (°)	5.36 to 61.14
Index ranges	-18 ≤ h ≤ 18, -10 ≤ k ≤ 10, -25 ≤ l ≤ 26
Reflections collected	18385
Independent reflections	2826 [R_{int} = 0.0335, R_{sigma} = 0.0257]
Data/restraints/parameters	2826/0/136
Goodness-of-fit on F^2	1.053
Final R indexes (I>=2σ (I))	R_1 = 0.0326, wR_2 = 0.0753
Final R indexes (all data)	R_1 = 0.0452, wR_2 = 0.0804
Largest diff. peak/hole ($e/Å^{-3}$)	0.51/-0.33

Preparation of various hydantoins and imides

(*S*)-Tetrahydro-1H-pyrrolo(1,2-c)imidazole-1,3(2H)-dione (1b) [CAS: 40856-87-9]

Hydantoin (**1b**) was prepared according to literature[2] with slight modifications to the procedure.



L-Proline (10.0 mmol, 1.0 eq), potassium cyanate (10.0 mmol, 1.0 eq) and water (3 mL) were added to a 10 mL round-bottom flask. The reaction mixture was refluxed for 1 h, and allowed to cool to room temp. Hydrochloric acid (35% (v/v), 1.5 mL) was added and the reaction was refluxed for an additional 15 min. The mixture was cooled in an ice bath and the crystals formed were filtered off and washed with cold water (2-3 mL). **1b** was obtained as colorless crystals (945.5 mg, 68%).

^{1}H NMR (600 MHz, DMSO-d_6): δ 10.74 (br s, 1H), 4.11 (dd, *J* = 9.0,7.6 Hz, 1H), 3.46 (dt, *J* = 10.8, 7.6 Hz, 1H), 3.04 (ddd, *J* = 10.8, 8.1, 4.7 Hz, 1H), 2.09 – 2.02 (m, 1H), 2.01 – 1.87 (m, 2H), 1.71 – 1.58 (m, 1H).

^{13}C NMR (151 MHz, DMSO-d_6): δ 175.4, 161.0, 64.0, 44.9, 26.7, 26.7.

HRMS (ESI) m/z [M + Na]$^{+}$: Calcd. for $C_6H_8N_2NaO_2^{+}$: 163.0478, found: 163.0478.

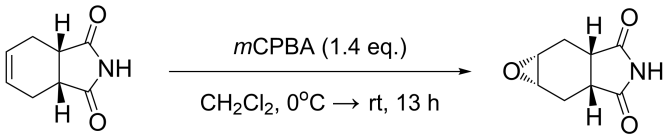
$(\alpha)_D^{20}$ = - 127.5 (c = 0.51, ethanol)

Melting point: 159 – 162^{o}C.

The spectroscopic and optical data is in accordance with the literature.[3]

(1aR,2aS,5aR,6aS)-hexahydro-3H-oxireno(2,3-f)isoindole-3,5(4H)-dione (1k) [CAS: 52918-43-1]

Epoxide (**1k**) was prepared according to literature[4].



cis-1,2,3,6-Tetrahydrophthalimide (0.757 g, 1.0 eq, 5 mmol) was dissolved in dry CH_2Cl_2 (30 mL) and *m*CPBA (1.78 g, 1.4 eq, 7 mmol) was added in small portions at 0°C while stirring. The mixture was allowed to warm to room temperature and stirred for 13 h. After addition of $CHCl_3$ (30 mL) and sat. aq. Na_2CO_3 (20 mL), the mixture was stirred for 30 min. Then the organic phase was separated, washed with sat. aq. Na_2CO_3 (3 x 10 mL), dried over Na_2SO_4 and the solvent was removed on a rotary evaporator. **1k** was isolated by recrystallization from methanol as colorless crystals (92.8 mg, 11%)

^{1}H NMR (600 MHz, CD_3OD): δ 3.18 – 3.16 (m, 2H), 2.86 – 2.79 (m, 2H), 2.60 – 2.53 (m, 2H), 2.25 – 2.16 (m, 2H).

^{13}C NMR (151 MHz, CD_3OD) δ 184.4, 52.1, 37.8, 23.0.

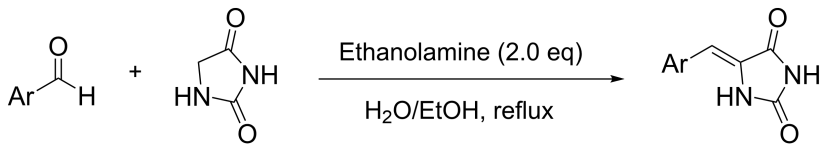
HRMS (ESI) m/z $[M + Na]^+$: Calcd. for $C_8H_9NNaO_3^+$: 190.0475, found: 190.0475.

The spectroscopic data is in accordance with the literature.[4]

Preparation of 5-arylidenehydantoins and 5-arylidene-2,4-thiazolidinedione

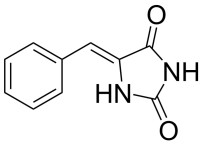
5-arylidenehydantoins (**1g**-**1j**) were prepared according to literature[5]

General procedure



Hydantoin (500 mg, 5.00 mmol, 1.0 eq) was added to a 250 mL round-bottom flask, dissolved in H_2O (50 mL) and placed in a heating block set to 70°C. Ethanolamine (0.61 mL, 10.11 mmol, 2.0 eq) was added to the mixture via a syringe, followed by dropwise addition of the appropriate aldehyde (5.02 mmol, 1.0 eq. in 5 mL ethanol). The mixture was heated at reflux (heating block was set to 110°C) for the time indicated. The mixture was allowed to cool down to room temperature, leading to precipitation of product. The precipitate was collected via vacuum filtration and washed with H_2O (3 x 10 mL) and ethanol (1-2 mL) and dried under high vacuum.

(*Z*)-5-Benzylidenehydantoin (1g) [CAS: 74805-60-0]



Condensation of benzaldehyde (0.51 mL, 5.02 mmol) by the general procedure for 6.5 h gave **1g** as a colorless solid (567.8 mg, 60%).

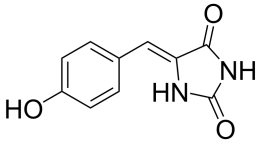
^{1}H NMR (600 MHz, DMSO-d_6): δ 11.19 (br s, 1H), 10.59 (br s, 1H), 7.63 – 7.56 (m, 2H), 7.41 – 7.37 (m, 2H), 7.35 – 7.29 (m, 1H), 6.42 (s, 1H).

^{13}C NMR (151 MHz, DMSO-d_6): δ 165.6, 155.8, 133.0, 129.4, 128.8, 128.4, 128.0, 108.4.

HRMS (ESI) m/z $[M + Na]^+$: Calcd. for $C_{10}H_8N_2NaO_2^+$: 211.0478, found: 211.0478.

The spectroscopic data is in accordance with the literature.[6]

(*Z*)-5-(4-Hydroxybenzylidene)hydantoin (1h) [CAS: 1134426-24-6]



Condensation of 4-hydroxybenzaldehyde (614.2 mg, 5.03 mmol) by the general procedure for 16 h gave **1h** as a yellow-green solid (274.6 mg, 27%).

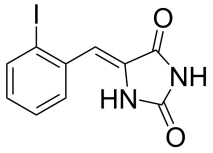
^{1}H NMR (600 MHz, DMSO-d_6): δ 11.08 (br s, 1H), 10.31 (br s, 1H), 9.87 (br s, 1H), 7.47 (d, *J* = 8.2 Hz, 2H), 6.79 (d, *J* = 8.2 Hz, 2H), 6.36 (s, 1H).

^{13}C NMR (151 MHz, DMSO-d_6): δ 165.7, 158.1, 155.7, 131.3, 125.4, 123.9, 115.8, 109.4.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for $C_{10}H_8N_2NaO_3^+$: 227.0427, found: 227.0426.

The spectroscopic data is in accordance with the literature.[7]

(*Z*)-5-(2-Iodobenzylidene)hydantoin (1i) [CAS: 896572-66-0]



Condensation of 2-iodobenzaldehyde (696.7 mg, 3.00 mmol) by the general procedure for 6.5 h gave **1i** as a colorless solid (285.1 mg, 30%).

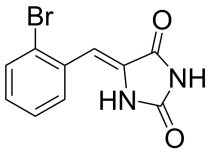
^{1}H NMR (600 MHz, DMSO-d_6): δ 11.28 (br s, 1H), 10.67 (br s, 1H), 7.94 (dd, *J* = 8.0, 1.3 Hz, 1H), 7.60 (dd, *J* = 7.8, 1.6 Hz, 1H), 7.43 (td, *J* = 7.6, 1.3 Hz, 1H), 7.07 (td, *J* = 7.6, 1.6 Hz, 1H), 6.41 (s, 1H).

^{13}C NMR (151 MHz, DMSO-d_6): δ 165.2, 155.6, 139.3, 135.9, 130.0, 129.8, 129.6, 128.7, 110.8, 101.6.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for $C_{10}H_7IN_2NaO_2^+$: 336.9444, found: 336.9444.

This compound exist in the literature, but without spectroscopic data.

(*Z*)-5-(2-Bromobenzylidene)hydantoin (1j) [CAS: 1331770-82-1]



Condensation of 2-bromobenzaldehyde (0.59 mL, 5.05 mmol) by the general procedure for 13 h gave **1j** as a colorless solid (638.6 mg, 48%).

^{1}H NMR (600 MHz, DMSO-d_6): δ 11.00 (br s, 2H), 7.68 (td, *J* = 7.8, 1.5 Hz, 2H), 7.41 (td, *J* = 7.5, 1.2 Hz, 1H), 7.26 (td, *J* = 7.7, 1.7 Hz, 1H), 6.51 (s, 1H).

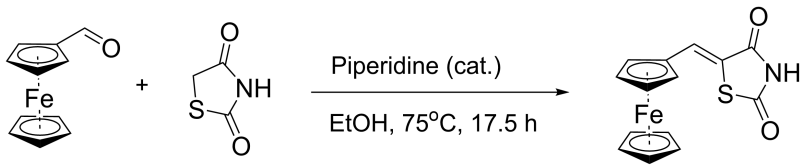
^{13}C NMR (151 MHz, DMSO-d_6): δ 165.2, 155.6, 132.9, 132.6, 130.2, 130.1, 130.0, 128.1, 124.1, 105.6.

HRMS (ESI) m/z [M + Na]$^{+}$: Calcd. for $C_{10}H_7{}^{79}BrN_2NaO_2^{+}$: 288.9583, found: 288.9583.

The spectroscopic data is in accordance with the literature.[8]

(*Z*)-5-Ferrocenyl-2,4-thiazolidinedione (1f) [CAS: 2234288-09-4]

(*Z*)-5-Ferrocenyl-2,4-thiazolidinedione (**1f**) was prepared according to literature[9]



Ferrocenecarboxaldehyde (1.070 g, 5.00 mmol, 1.0 eq) and 2,4-thiazolidinedione (626.7 mg, 5.35 mmol, 1.07 eq) were dissolved in ethanol (20 mL), followed by addition of a catalytic amount of piperidine (~ 10 drops). The reaction mixture was stirred at 75°C overnight (17.5 h) and then allowed to cool to room temperature until a precipitate was formed. The precipitate was collected by filtration, washed with water (3 x 2 mL) and dried under high vacuum to afford compound **1f** as a wine-red solid (1.088 g, 63%).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.75 (s, 1H), 4.59 – 4.56 (m, 4H), 4.23 (s, 5H).

^{13}C NMR (151 MHz, $CDCl_3$): δ 167.1, 165.8, 137.1, 117.9, 76.1, 72.4, 70.7, 70.1.

HRMS (ESI) m/z [M + Na]$^{+}$: Calcd. for $C_{14}H_{11}FeNO_2S^{+}$: 312.9854, found: 312.9854.

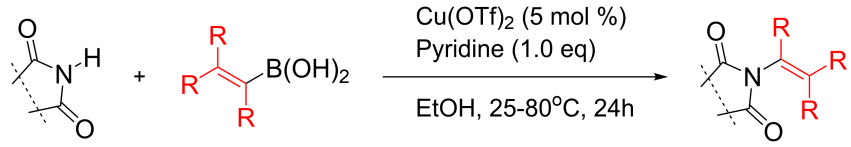
The spectroscopic data is in accordance with the literature.[9]

Preparation of alkenylboronic acids

(*E*)-(2-(9-phenanthrenyl)vinyl)boronic acid (**2d**) was prepared according to literature[10]

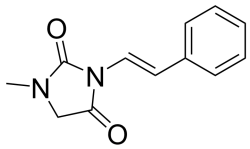
Synthesis of *N*-alkenylimides

General procedure



Imide (0.40 mmol, 1.0 eq), alkenylboronic acid (0.40-0.60 mmol, 2.0-3.0 eq), $Cu(OTf)_2$ (7.2 mg, 0.020 mmol, 0.050 eq) and ethanol (2 mL) were added to a 5 mL round-bottom flask equipped with a magnetic stir bar. Pyridine (32 µL, 0.40 mmol, 1.0 eq) was added via a syringe, a cooler was connected to the flask and the mixture was stirred with access to air at the indicated temperature (25-80°C) for 24 hours. The crude mixture was either purified by filtration followed by washing, column chromatography (using silica gel and eluent system as specified) or both as specified. The desired product was dried under high vacuum.

(*E*)-1-Methyl-3-styrylhydantoin (3a) [NEW]



Following the general procedure at 25°C, **3a** was obtained after column chromatography (chloroform:hexane:acetone [7:2:1]) as a colorless solid (84.8 mg, 98 %).

R_F = 0.39 (chloroform:hexane:acetone [7:2:1]).

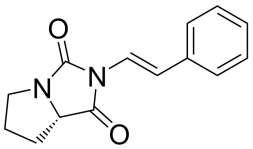
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.54 (d, *J* = 15.2 Hz, 1H), 7.42 – 7.37 (m, 2H), 7.33 – 7.28 (m, 2H), 7.25 – 7.20 (m, 1H), 7.11 (d, *J* = 15.1 Hz, 1H), 3.84 (s, 2H), 2.99 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$): δ 167.5, 154.6, 135.6, 128.6, 127.4, 126.0, 119.5, 118.0, 50.7, 29.6.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for $C_{12}H_{12}N_2NaO_2^+$: 239.0791, found: 239.0791.

FT-IR (neat, ν_{max} cm^{-1}): 3049, 2937, 1715, 1647, 1382, 955.

(*S*,*E*)-2-styryltetrahydro-1H-pyrrolo(1,2-c)imidazole-1,3(2H)-dione (3b) [NEW]



Following the general procedure at 25°C, **3b** was obtained after column chromatography (chloroform:hexane:acetone [8:1:1]) as an off-white solid (94.4 mg, 97 %).

R_F = 0.42 (chloroform:hexane:acetone [8:1:1]).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.58 (d, *J* = 15.1 Hz, 1H), 7.45 – 7.41 (m, 2H), 7.37 – 7.33 (m, 2H), 7.29 – 7.25 (m, 1H), 7.11 (d, *J* = 15.1 Hz, 1H), 4.16 (dd, *J* = 9.2, 7.5 Hz, 1H), 3.78 (dt, *J* = 11.3, 7.7 Hz, 1H), 3.34 (ddd, *J* = 11.3, 8.3, 4.7 Hz, 1H), 2.34 (dtd, *J* = 12.7, 7.3, 3.7 Hz, 1H), 2.21 – 2.06 (m, 2H), 1.81 (dtd, *J* = 12.7, 9.4, 8.3 Hz, 1H).

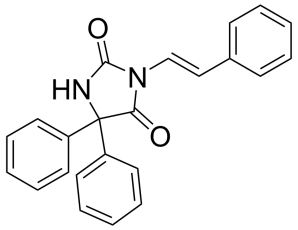
^{13}C NMR (151 MHz, $CDCl_3$): δ 171.6, 158.3, 135.6, 128.6, 127.6, 126.1, 120.2, 118.0, 62.5, 45.6, 27.6, 26.8.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for $C_{14}H_{14}N_2NaO_2^+$: 265.0947, found: 265.0947.

$(\alpha)_D^{20}$ = - 66.1 (c = 0.56, $CHCl_3$).

FT-IR (neat, ν_{max} cm^{-1}): 3053, 2980, 2894, 1764, 1699, 952, 749.

(*E*)-5,5,-diphenyl-3-styrylhydantoin (3c) [NEW]



Following the general procedure at 25°C, **3c** was obtained after filtration and washing with ethanol (3 x 1 mL) of the crude reaction mixture as a colorless solid (121.5 mg, 86 %).

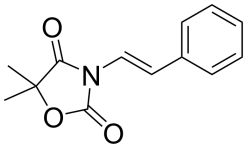
^{1}H NMR (600 MHz, Acetone-d_6): δ 8.83 (br s, 1H), 7.63 (d, *J* = 15.1 Hz, 1H), 7.51 – 7.48 (m, 6H), 7.46 – 7.42 (m, 4H), 7.42 – 7.38 (m, 2H), 7.38 – 7.33 (m, 2H), 7.28 – 7.25 (m, 1H), 7.24 (d, *J* = 15.1 Hz, 1H).

^{13}C NMR (151 MHz, Acetone-d_6): δ 172.3, 154.3, 140.6, 136.7, 129.6, 129.5, 129.3, 128.5, 127.9, 126.9, 120.6, 119.1, 69.8.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for $C_{23}H_{18}N_2NaO_2^+$: 377.1260, found: 377.1260.

FT-IR (neat, ν_{max} cm^{-1}): 3170, 3060, 2995, 1735, 1712, 953.

(*E*)-3-Styryl-5,5-dimethyl-2,4-oxazolidinedione (3d) [NEW]



Following the general procedure at 25°C, **3d** was obtained after column chromatography (hexane:acetone [9:1]) as a pale yellow oil (92.0 mg, 100 %).

R_F = 0.23 (hexane:acetone [9:1]).

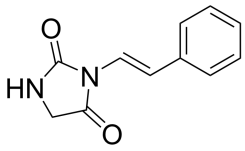
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.57 (d, *J* = 15.1 Hz, 1H), 7.43 – 7.40 (m, 2H), 7.37 – 7.33 (m, 2H), 7.30 – 7.27 (m, 1H), 7.04 (d, *J* = 15.1 Hz, 1H), 1.63 (s, 6H).

^{13}C NMR (151 MHz, $CDCl_3$): δ 173.9, 152.3, 134.7, 128.8, 128.2, 126.3, 122.1, 117.0, 82.7, 23.7.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for $C_{13}H_{13}NNaO_3^+$: 254.0788, found: 254.0787.

FT-IR (neat, ν_{max} cm^{-1}): 3067, 3027, 2994, 1816, 1731, 1652, 1382, 963.

(*E*)-3-Styrylhydantoin (3e) [NEW]



<u>0.40 mmol scale:</u> Following the general procedure at 25°C using 2 equivalents of (*E*)-styrylboronic acid, **3e** was obtained after column chromatography (chloroform:acetonitrile [9:2]) as a colorless solid (62.5 mg, 77 %).

<u>1.5 mmol scale:</u> Hydantoin (149.8 mg, 1.5 mmol, 1.0 eq), (*E*)-styrylboronic acid (444.7 mg, 3.0 mmol, 2.0 eq), $Cu(OTf)_2$ (26.6 mg, 0.075 mmol, 0.050 eq) and ethanol (7 mL) were added to a 25 mL round-bottom flask equipped with a magnetic stir bar. Pyridine (120 µL, 1.5 mmol, 1.0 eq) was added via a syringe, a cooler was connected to the flask and the mixture was stirred at 25°C for 24 hours. **3e** was obtained after column chromatography (chloroform:acetonitrile [9:2]) as a colorless solid (267.6 mg, 88 %).

R_F = 0.31 (chloroform:acetonitrile [9:2]).

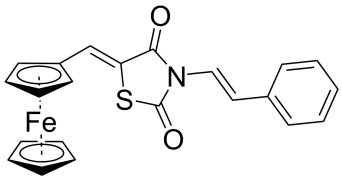
^{1}H NMR (600 MHz, DMSO-d_6): δ 8.45 (br s, 1H), 7.46 – 7.41 (m, 3H (overlap with vinylic proton)), 7.34 (t, *J* = 7.8 Hz, 2H), 7.26 – 7.22 (m, 1H), 7.09 (d, *J* = 15.2 Hz, 1H), 3.99 (d, *J* = 1.0 Hz, 2H).

^{13}C NMR (151 MHz, DMSO-d_6): δ 170.4, 155.5, 135.7, 128.8, 127.4, 125.8, 118.5, 118.1, 45.3.

HRMS (ESI) m/z $[M + Na]^+$: Calcd. for $C_{11}H_{10}N_2NaO_2^+$: 225.0634, found: 225.0635.

FT-IR (neat, ν_{max} cm^{-1}): 3232, 3074, 2921, 1781, 1700, 962.

((*Z*)-5-ferrocenylidene)-((*E*)-3-styryl)-2,4-thiazolidinedione (3f) [NEW]



Following the general procedure at 25°C**, 3f** was obtained after filtration and washing with ethanol (3 x 1 mL) of the crude reaction mixture as a wine-red solid (156.4 mg, 94 %).

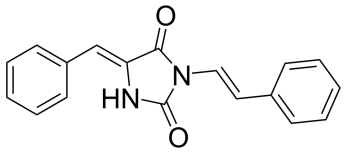
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.85 (s, 1H), 7.68 (d, *J* = 15.0 Hz, 1H), 7.49 – 7.45 (m, 2H), 7.36 (t, *J* = 7.6 Hz, 2H), 7.31 (d, *J* = 15.0 Hz, 1H), 7.30 – 7.27 (m, 1H), 4.62 – 4.58 (m, 4H), 4.23 (s, 5H).

^{13}C NMR (151 MHz, $CDCl_3$): δ 166.4, 164.0, 137.4, 135.4, 128.7, 128.0, 126.5, 122.7, 119.1, 115.4, 76.4, 72.4, 70.8, 70.1.

HRMS (ESI) m/z $[M + Na]^+$: Calcd. for $C_{22}H_{17}FeNNaO_2S^+$: 438.0222, found: 438.0221.

FT-IR (neat, ν_{max} cm^{-1}): 3083, 3036, 1731, 1670. 1602, 961, 822.

(*Z*)-5-Benzylidene-(*E*)-3-styrylhydantoin (3g) [NEW]



Following the general procedure at 40°C, **3g** was obtained after filtration and washing with ethanol (3 x 0.5 mL) of the crude reaction mixture and column chromatography (chloroform:hexane:acetone [7:2:1]) as a colorless solid (91.4 mg, 80 %).

R_F = 0.48 (chloroform:hexane:acetone [7:2:1]).

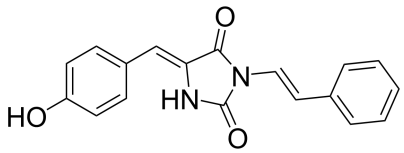
^{1}H NMR (600 MHz, DMSO-d_6): δ 11.13 (br s, 1H), 7.68 (d, *J* = 7.6 Hz, 2H), 7.50 (d, *J* = 7.6 Hz, 2H), 7.47 (d, *J* = 15.2 Hz, 1H), 7.43 (t, *J* = 7.6 Hz, 2H), 7.37 (t, *J* = 7.9 Hz, 3H), 7.27 (t, *J* = 7.4 Hz, 1H), 7.19 (d, *J* = 15.1 Hz, 1H), 6.64 (s, 1H).

^{13}C NMR (151 MHz, DMSO-d_6): δ 162.3, 153.2, 135.4, 132.6, 129.7, 128.9 (2C), 128.8, 127.6, 126.0, 125.5, 119.2, 118.0, 110.6.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for $C_{18}H_{14}N_2NaO_2^+$: 313.0947, found: 313.0947.

FT-IR (neat, ν_{max} cm^{-1}): 3193, 3031, 1767, 1718, 1704, 1658, 958, 833.

(*Z*)-(5-(4-hydroxybenzylidene))-(*E*)-3-styrylhydantoin (3h) [NEW]



Following the general procedure at 25°C**,** **3h** was purified by column chromatography (chloroform:acetone [9:4]). The collected fractions were concentrated under reduced pressure, the resulting solid was transferred to a fritted funnel and washed with $CHCl_3$ (3 x ~1 mL). **3h** was obtained as a pale yellow solid (102.8 mg, 84 %).

R_F = 0.51 (chloroform:acetone [9:4]).

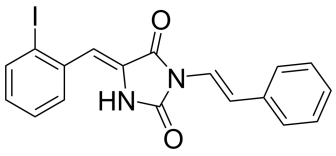
^{1}H NMR (600 MHz, DMSO-d_6): δ 10.92 (br s, 1H), 9.96 (br s, 1H), 7.55 (d, *J* = 8.7 Hz, 1H), 7.50 – 7.45 (m, 3H), 7.36 (t, *J* = 7.6 Hz, 2H), 7.28 – 7.24 (m, 1H), 7.19 (d, *J* = 15.1 Hz, 1H), 6.82 (d, *J* = 8.6 Hz, 2H), 6.58 (s, 1H).

^{13}C NMR (151 MHz, DMSO-d_6): δ 162.4, 158.5, 153.1, 135.5, 131.7, 128.9, 127.6, 125.9, 123.6, 122.7, 119.0, 118.1, 115.8, 111.8.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for $C_{18}H_{14}N_2NaO_3^+$: 329.0897, found: 329.0896.

FT-IR (neat, ν_{max} cm^{-1}): 3325, 3206, 3078, 1746, 1704, 1699, 1404, 958, 826.

(*Z*)-(5-(2-iodobenzylidene))-(*E*)-3-styrylhydantoin (3i) [NEW]



Following the general procedure at 25°C, **3i** was obtained after filtration and washing with ethanol (3 x 0.5 mL) of the crude reaction mixture and column chromatography (chloroform:hexane:acetone [7:2:1]) as a pale yellow solid (122.0 mg, 73 %).

R_F = 0.47 (chloroform:hexane:acetone [7:2:1]).

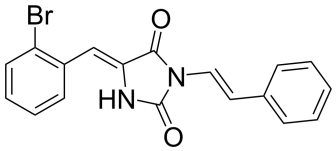
^{1}H NMR (600 MHz, DMSO-d_6): δ 11.24 (br s, 1H), 7.97 (dd, *J* = 7.9, 1.2 Hz, 1H), 7.64 (dd, *J* = 7.7, 1.6 Hz, 1H), 7.51 – 7.48 (m, 2H), 7.47 (d, *J* = 15.1 Hz, 1H), 7.46 (td, *J* = 7.6, 1.2 Hz, 1H), 7.38 – 7.35 (m, 2H), 7.29 – 7.25 (m, 1H), 7.17 (d, *J* = 15.0 Hz, 1H), 7.10 (td, *J* = 7.6, 1.6 Hz, 1H), 6.60 (s, 1H).

^{13}C NMR (151 MHz, DMSO-d_6): δ 162.0, 153.1, 139.4, 135.6, 135.4, 130.4, 129.9, 128.9, 128.7, 127.7, 127.5, 126.0, 119.4, 117.9, 113.1, 101.8.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for $C_{18}H_{13}IN_2NaO_2^+$: 438.9914, found: 438.9913.

FT-IR (neat, ν_{max} cm^{-1}): 3232, 3024, 1754, 1713, 1643, 957.

(*Z*)-(5-(2-bromobenzylidene))-(*E*)-3-styrylhydantoin (3j) [NEW]



Following the general procedure at 25°C, **3j** was obtained after filtration and washing with ethanol (3 x 0.5 mL) of the crude reaction mixture and column chromatography (chloroform:hexane:acetone [7:2:1]) as a pale yellow solid (124.2 mg, 84 %).

R_F = 0.44 (chloroform:hexane:acetone [7:2:1]).

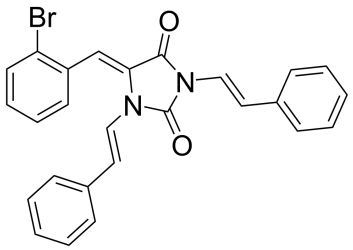
^{1}H NMR (600 MHz, DMSO-d_6): δ 11.27 (br s, 1H), 7.73 – 7.70 (m, 2H), 7.51 – 7.48 (m, 2H), 7.48 – 7.42 (m, 2H), 7.38 – 7.35 (m, 2H), 7.32 – 7.28 (m, 1H), 7.29 – 7.25 (m, 1H), 7.17 (d, *J* = 15.1 Hz, 1H), 6.70 (s, 1H).

^{13}C NMR (151 MHz, DMSO-d_6): δ 162.0, 153.1, 135.3, 132.9, 132.3, 130.5, 130.4, 128.9, 128.1, 127.8, 127.7, 126.0, 124.2, 119.4, 117.9, 107.9.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for $C_{18}H_{13}{}^{79}BrN_2NaO_2^+$: 391.0053, found: 391.0053

FT-IR (neat, ν_{max} cm^{-1}): 3237, 3071, 3025, 1751, 1718, 1644, 955, 670, 638.

(*Z*)-(5-(2-bromobenzylidene))-(E,E)-1,3-distyrylhydantoin (3j') [NEW]



Following the general procedure at 25°C, **3j'** was obtained (as a biproduct in the isolation of compound **3j**) after filtration and washing with ethanol (3 x 0.5 mL) of the crude reaction mixture and column chromatography (chloroform:hexane:acetone [7:2:1]) as a yellow solid (30.4 mg, 16 %).

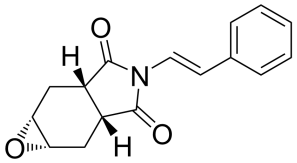
R_F = 0.75 (chloroform:hexane:acetone [7:2:1]).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.70 (d, *J* = 15.1 Hz, 1H), 7.66 – 7.61 (m, 1H), 7.49 – 7.46 (m, 2H), 7.37 (t, *J* = 7.7 Hz, 2H), 7.32 – 7.27 (m, 2H), 7.26 (d, *J* = 15.1 Hz, 1H), 7.25 – 7.17 (m, 6H), 7.16 (s, 1H), 6.93 – 6.90 (m, 2H), 6.58 (d, *J* = 14.7 Hz, 1H), 6.51 (d, *J* = 14.7 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$): δ 160.5, 151.5, 135.4, 134.7, 132.7, 131.6, 130.3, 128.8, 128.5, 127.9, 127.7, 126.9, 126.9, 126.3, 125.9, 124.5, 123.6, 121.4, 119.6, 117.1.

FT-IR (neat, ν_{max} cm^{-1}): 3023, 2968, 1766, 1725, 956, 836, 659.

(1a*R*,2a*S*,5a*R*,6a*S*)-4-((*E*)-styryl)hexahydroazirino(2,3-*f*)isoindole-3,5(1*H*,4*H*)-dione (3k) [NEW]



Following the general procedure at 25°C, **3k** was obtained after filtration and washing with ethanol (3 x 1 mL) of the crude reaction mixture as a colorless solid (94.5 mg, 91 %).

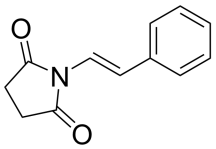
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.63 (d, *J* = 15.2 Hz, 1H), 7.44 – 7.40 (m, 2H), 7.34 – 7.29 (m, 2H), 7.25 – 7.22 (m, 1H), 7.18 (d, *J* = 15.1 Hz, 1H), 3.16 (dt, *J* = 4.5, 2.2 Hz, 2H), 2.82 – 2.77 (m, 2H), 2.77 – 2.74 (m, 1H), 2.74 – 2.71 (m, 1H), 2.22 – 2.15 (m, 2H).

^{13}C NMR (151 MHz, $CDCl_3$): δ 178.5, 135.7, 128.5, 127.6, 126.2, 121.3, 118.2, 50.5, 34.8, 22.5.

HRMS (ESI) m/z $[M + Na]^+$: Calcd. for $C_{16}H_{15}NNaO_3^+$: 292.0944, found: 292.0944.

FT-IR (neat, ν_{max} cm^{-1}): 3074, 2933, 2858, 1701, 958.

(*E*)-*N*-Styrylsuccinimide (3l) [CAS: 120542-02-1]



Following the general procedure at 25°C, **3l** was obtained after filtration and washing with ethanol (3 x 1 mL) of the crude reaction mixture as a pale, grey-brown solid (72.0 mg, 90 %).

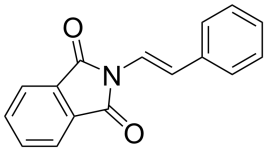
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.68 (d, *J* = 15.2 Hz, 1H), 7.46 – 7.41 (m, 2H), 7.37 – 7.31 (m, 3H), 7.29 – 7.24 (m, 1H), 7.20 (d, *J* = 15.2 Hz, 1H), 2.79 (s, 4H).

^{13}C NMR (151 MHz, $CDCl_3$): δ 175.2, 135.5, 128.7, 127.9, 126.4, 122.0, 117.8, 27.8.

HRMS (ESI) m/z $[M + Na]^+$: Calcd. for $C_{12}H_{11}NNaO_2^+$: 224.0682, found: 224.0682.

The spectroscopic data is in accordance with the literature.[11]

(*E*)-*N*-Styrylphthalimide (3m) [CAS: 32150-95-1]



<u>0.4 mmol scale</u>: Following the general procedure at 25°C, **3m** was obtained after filtration and washing with ethanol (3 x 1 mL) of the crude reaction mixture as a yellow solid (94.1 mg, 95 %).

<u>1.0 mmol scale:</u> Phthalimide (147.2 mg, 1.0 mmol, 1.0 eq), (*E*)-styrylboronic acid (443.3 mg, 3.0 mmol, 3.0 eq), $Cu(OTf)_2$ (18.3 mg, 0.050 mmol, 0.050 eq) and ethanol (5 mL) were added to a 10 mL round-bottom flask equipped with a magnetic stir bar. Pyridine (80 µL, 1.0 mmol, 1.0 eq) was added via a syringe, a cooler was connected to the flask and the mixture was stirred at 25°C for 24 hours. **3m** was obtained after filtration and washing with ethanol (3 x 2 mL) of the crude reaction mixture as a yellow solid (235.6 mg, 95 %).

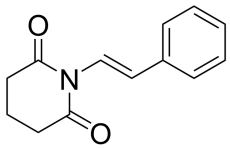
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.88 (dd, *J* = 5.4, 3.1 Hz, 2H), 7.74 (dd, *J* = 5.5, 3.0 Hz, 2H), 7.64 (d, *J* = 15.2 Hz, 1H), 7.46 (d, *J* = 7.2 Hz, 2H), 7.38 – 7.30 (m, 3H (overlap with vinylic proton)), 7.28 – 7.22 (m, 1H).

^{13}C NMR (151 MHz, $CDCl_3$): δ 166.3, 135.9, 134.4, 131.6, 128.6, 127.5, 126.1, 123.6, 120.1, 117.5.

HRMS (ESI) m/z $[M + Na]^+$: Calcd. for $C_{16}H_{11}NNaO_2^+$: 272.0682 , found: 272.0681.

The spectroscopic data is in accordance with the literature[12]

(*E*)-*N*-Styrylglutarimide (3p) [NEW]



Following the general procedure at 25°C, **3p** was obtained after column chromatography (chloroform:hexane:acetone [18:1:1]) as a colorless solid (77.2 mg, 90 %).

R_F = 0.45 (chloroform:hexane:acetone [18:1:1]).

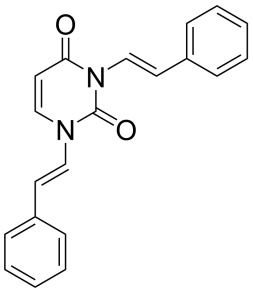
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.43 – 7.40 (m, 2H), 7.34 – 7.30 (m, 2H), 7.27 – 7.23 (m, 1H), 7.15 (ABq, *J* = 15.0 Hz, 2H), 2.76 (t, *J* = 6.5 Hz, 4H), 2.00 (p, *J* = 6.6 Hz, 2H).

^{13}C NMR (151 MHz, $CDCl_3$): δ 171.8, 135.7, 128.6, 127.8, 127.5, 126.5, 120.0, 33.5, 16.8.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for $C_{13}H_{13}NNaO_2^+$: 238.0838, found: 238.0838.

FT-IR (neat, ν_{max} cm^{-1}): 3026, 2959, 1729, 1664, 953.

(*E*,*E*)-1,3-distyryluracil (3q) [NEW]



Following the general procedure at 25°C using 4 equivalents of (*E*)-styrylboronic acid, **3q** was obtained after filtration and washing with ethanol (3 x 1 mL) of the crude reaction mixture as a colorless solid (109.4 mg, 86 %).

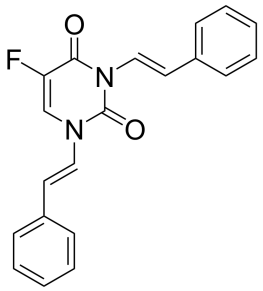
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.66 (d, *J* = 14.7 Hz, 1H), 7.59 (d, *J* = 8.1 Hz, 1H), 7.52 – 7.46 (m, 3H), 7.45 – 7.40 (m, 2H), 7.40 – 7.33 (m, 5H), 7.33 – 7.30 (m, 1H), 7.30 – 7.27 (m, 1H), 6.56 (d, *J* = 14.7 Hz, 1H), 5.95 (d, *J* = 8.1 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$): δ 161.5, 149.3, 137.5, 135.6, 134.1, 128.9, 128.6, 128.4, 128.0, 127.9, 126.7, 126.5, 124.4, 120.2, 119.9, 103.0.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for $C_{20}H_{16}N_2NaO_2^+$: 339.1104, found: 339.1103.

FT-IR (neat, ν_{max} cm^{-1}): 3103, 3028, 1724, 1640, 960, 693.

(*E*,*E*)-1,3-distyryl-5-fluorouracil (3r) [NEW]



Following the general procedure at 25°C using 4 equivalents of (*E*)-styrylboronic acid, **3r** was obtained after filtration and washing with ethanol (3 x 1 mL) of the crude reaction mixture as a colorless solid (107.3 mg, 80 %).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.70 (d, *J* = 5.3 Hz, 1H), 7.67 (dd, *J* = 14.7, 1.7 Hz ($^{5}J_{H\text{-}F}$), 1H), 7.50 (t, *J* = 14.9 Hz, 1H), 7.49 – 7.45 (m, 2H), 7.44 – 7.40 (m, 2H), 7.38 (d, *J* = 7.6 Hz, 2H), 7.37 – 7.34 (m, 2H), 7.34 – 7.29 (m, 3H), 6.50 (d, *J* = 14.7 Hz, 1H).

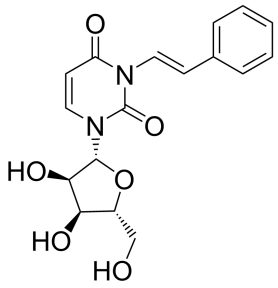
^{13}C NMR (151 MHz, $CDCl_3$): δ 155.9 (d, $J_{C\text{-}F}$ = 25.8 Hz), 147.7, 140.4 (d, $J_{C\text{-}F}$ = 237.7 Hz), 135.1, 133.8, 129.0, 128.8, 128.7, 128.5, 128.4, 126.8, 126.4, 123.8, 122.0 (d, $J_{C\text{-}F}$ = 34.6 Hz), 119.8, 119.5.

^{19}F NMR (376 MHz, $CDCl_3$): δ -161.5.

HRMS (ESI) m/z $[M + Na]^+$: Calcd. for $C_{20}H_{15}FN_2NaO_2^+$: 357.1010, found: 357.1009.

FT-IR (neat, ν_{max} cm^{-1}): 3070, 3024, 1712, 1682, 1664, 978, 958, 687.

(*E*)-3-styryluridine (3s) [NEW]



Following the general procedure at 25°C, **3s** was obtained after column chromatography. Two consecutive columns were necessary for the purification of **3s** with acetone as eluent in the first column and acetonitrile as eluent in the second column. The desired fractions were collected and the solvent was removed after each column before the mixture of product and impurities was re-applied on to the next column. **3s** was obtained as an off-white solid (51.0 mg, 37 %). *Note: product tailing on the columns*.

R_F = 0.32 (acetone). *Note: The product was not visible in UV-light on TLC when the crude reaction mixture was analyzed, but visible after isolation*

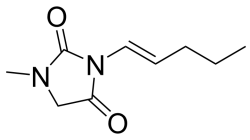
^{1}H NMR (600 MHz, CD_3CN): δ 7.89 (d, *J* = 8.1 Hz, 1H), 7.51 – 7.42 (m, 2H), 7.36 (t, *J* = 7.6 Hz, 2H), 7.33 (d, *J* = 14.8 Hz, 1H), 7.31 – 7.27 (m, 1H), 7.26 (d, *J* = 14.8 Hz, 1H), 5.85 (d, *J* = 3.9 Hz, 1H), 5.75 (d, *J* = 8.2 Hz, 1H), 4.17 (q, *J* = 4.5 Hz, 1H), 4.14 (q, *J* = 5.2 Hz, 1H), 3.97 (dt, *J* = 5.5, 2.8 Hz, 1H), 3.87 – 3.77 (m, 2H), 3.70 (ddd, *J* = 12.3, 5.1, 2.9 Hz, 1H), 3.53 (d, *J* = 5.9 Hz, 1H), 3.32 (t, *J* = 5.1 Hz, 1H).

^{13}C NMR (151 MHz, CD_3CN): δ 163.0, 151.6, 139.7, 136.8, 129.8, 128.9, 128.0, 127.3, 122.0, 102.0, 91.3, 85.6, 75.5, 70.5, 61.7.

HRMS (ESI) m/z $[M + Na]^+$: Calcd. for $C_{17}H_{18}N_2NaO_6^+$: 369.1057, found: 369.1057.

FT-IR (neat, ν_{max} cm^{-1}): 3375, 3078, 2946, 2823, 1696, 1635, 1455, 1402, 1106, 1074, 966, 695.

(*E*)-1-Methyl-3-(1,1-pentenyl)hydantoin (4a) [NEW]



Following the general procedure at 80°C, **4a** was obtained after column chromatography (chloroform:hexane:acetone [8:1:1]) as a yellow oil (57.6 mg, 79 %).

R_F = 0.39 (chloroform:hexane:acetone [8:1:1]).

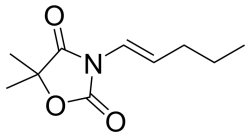
^{1}H NMR (600 MHz, $CDCl_3$): δ 6.49 (dt, *J* = 14.6, 7.2 Hz, 1H), 6.38 (dt, *J* = 14.7, 1.4 Hz, 1H), 3.85 (s, 2H), 3.01 (s, 3H), 2.08 (qd, *J* = 7.3, 1.4 Hz, 2H), 1.45 (sext, *J* = 7.4 Hz, 2H), 0.91 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$): δ 167.8, 155.3, 122.3, 118.1, 51.0, 32.9, 29.7, 22.5, 13.6.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for $C_9H_{14}N_2NaO_2^+$: 205.0947, found: 205.0947.

FT-IR (neat, ν_{max} cm^{-1}): 2920, 2862, 1735, 1705, 1375, 953.

(*E*)-3-(1,1-pentenyl)-5,5-dimethyl-2,4-oxazolidinedione (4b) [NEW]



Following the general procedure at 80°C, **4b** was obtained after column chromatography (toluene) as a colorless oil (52.1 mg, 66 %).

R_F = 0.42 (toluene).

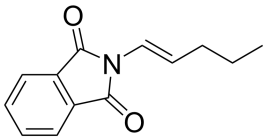
^{1}H NMR (600 MHz, $CDCl_3$): δ 6.53 (dt, *J* = 14.6, 7.3 Hz, 1H), 6.32 (dt, *J* = 14.6, 1.5 Hz, 1H), 2.10 (qd, *J* = 7.3, 1.5 Hz, 2H), 1.58 (s, 6H), 1.47 (sext, *J* = 7.4 Hz, 2H), 0.93 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$): δ 174.2, 152.7, 124.6, 117.3, 82.5, 32.7, 23.6, 22.2, 13.6.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for $C_{10}H_{15}NNaO_3^+$: 220.0950, found: 220.0949.

FT-IR (neat, ν_{max} cm^{-1}): 2948, 1735, 1672, 2959, 1382, 961.

(*E*)-*N*-(1,1-pentenyl)phthalimide (4c) [CAS: 2389180-23-6]



Following the general procedure at 80°C, **4c** was obtained after column chromatography (chloroform:hexane:acetone [7:4:1]) as a yellow crystalline solid (76.1 mg, 88 %).

R_F = 0.60 (chloroform:hexane:acetone [7:4:1]).

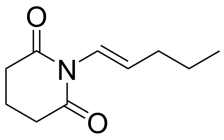
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.86 – 7.82 (m, 2H), 7.74 – 7.69 (m, 2H), 6.62 – 6.54 (m, 2H), 2.17 – 2.12 (m, 2H), 1.49 (h, *J* = 7.3 Hz, 2H), 0.95 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$): δ 166.7, 134.2, 131.7, 123.4, 122.7, 117.6, 33.2, 22.6, 13.6.

HRMS (ESI) m/z $[M + Na]^+$: Calcd. for $C_{13}H_{13}NNaO_2^+$: 238.0838, found: 238.0839.

The spectroscopic data is in accordance with the literature.[13]

(*E*)-*N*-(1,1-pentenyl)glutarimide (4d) [NEW]



Following the general procedure at 80°C using 2.5 equivalents of (*E*)-1-pentenylboronic acid, **4d** was obtained after column chromatography (chloroform:hexane:acetone [7:1:1]) as a pale yellow oil (40.6 mg, 56 %).

R_F = 0.45 (chloroform:hexane:acetone [7:1:1]).

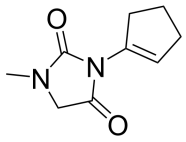
^{1}H NMR (600 MHz, $CDCl_3$): δ 6.19 (dt, *J* = 14.4, 1.6 Hz, 1H), 5.94 (dt, *J* = 14.4, 7.2 Hz, 1H), 2.67 (t, *J* = 6.6 Hz, 4H), 2.11 (qd, *J* = 7.3, 1.5 Hz, 2H), 1.93 (p, *J* = 6.6 Hz, 2H), 1.44 (sext, *J* = 7.4 Hz, 2H), 0.91 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$): δ 172.0, 131.1, 119.8, 33.2, 32.6, 22.1, 16.9, 13.5.

HRMS (ESI) m/z $[M + Na]^+$: Calcd. for $C_{10}H_{15}NNaO_2^+$: 204.0995, found: 204.0995.

FT-IR (neat, ν_{max} cm^{-1}): 2960, 2872, 1733, 1679, 1463, 1363, 951.

3-(1,1-cyclopentenyl)-1-methylhydantoin (4e) [NEW]



Following the general procedure at 80°C, **4e** was obtained after column chromatography (chloroform:hexane:acetone [7:2:1]) as a yellow wax (41.5 mg, 57 %).

R_F = 0.32 (chloroform:hexane:acetone [7:2:1]).

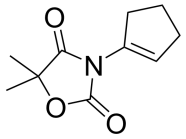
^{1}H NMR (600 MHz, $CDCl_3$): δ 6.02 – 5.97 (m, 1H), 3.88 (s, 2H), 3.00 (s, 3H), 2.77 – 2.71 (m, 2H), 2.48 – 2.42 (m, 2H), 1.99 – 1.92 (m, 2H).

^{13}C NMR (151 MHz, $CDCl_3$): δ 168.2, 155.5, 132.7, 123.9, 51.3, 31.6, 30.4, 29.7, 21.7.

HRMS (ESI) m/z $[M + Na]^+$: Calcd. for $C_9H_{12}N_2NaO_2^+$: 203.0791, found: 203.0791

FT-IR (neat, ν_{max} cm^{-1}): 3082, 2916, 1697, 1371, 806.

3-(1,1-cyclopentenyl)-5,5-dimethyl-2,4-oxazolidinedione (4g) [NEW]



Following the general procedure at 80°C, **4g** was obtained after column chromatography (toluene) as a pale yellow oil (61.3 mg, 79 %). *Note: Product tailing on the column*.

R_F = 0.45 (toluene).

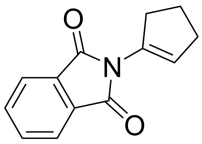
^{1}H NMR (600 MHz, $CDCl_3$): δ 6.11 – 6.08 (m, 1H), 2.78 – 2.73 (m, 2H), 2.47 – 2.41 (m, 2H), 2.00 – 1.92 (m, 2H), 1.56 (s, 6H).

^{13}C NMR (151 MHz, $CDCl_3$): δ 174.5, 152.6, 131.8, 124.3, 82.7, 31.2, 30.3, 23.7, 21.6.

HRMS (ESI) m/z $[M + Na]^+$: Calcd. for $C_{10}H_{13}NNaO_3^+$: 218.0788, found: 218.0787.

FT-IR (neat, ν_{max} cm^{-1}): 2937, 1818, 1736, 1597, 1382.

***N*-(1,1-cyclopentenyl)phthalimide (4h) [NEW]**



Following the general procedure at 80°C, **4h** was obtained after column chromatography (chloroform:hexane:acetone [7:4:1]) as a yellow crystalline solid (21.4 mg, 25 %).

R_F = 0.64 (chloroform:hexane:acetone [7:4:1]).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.85 (dd, *J* = 5.5, 3.1 Hz, 2H), 7.72 (dd, *J* = 5.5, 3.0 Hz, 2H), 6.08 – 6.06 (m, 1H), 2.86 – 2.79 (m, 2H), 2.54 – 2.47 (m, 2H), 2.05 – 1.98 (m, 2H).

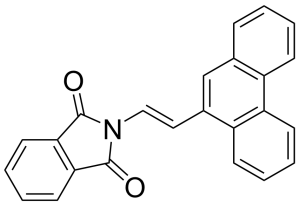
^{13}C NMR (151 MHz, $CDCl_3$): δ 167.0, 134.1, 132.7, 131.9, 124.1, 123.4, 32.1, 30.5, 21.9.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for $C_{13}H_{11}NNaO_2^+$: 236.0682, found: 236.0682.

FT-IR (neat, ν_{max} cm^{-1}): 3037, 2906, 1730, 1709, 818.

Melting point: 134 – 135°C.

(*E*)-*N*-(2-(9-phenanthrenyl)vinyl)phthalimide (4i) [NEW]



Following the general procedure at a 0.18 mmol scale at 25°C, **4i** was obtained after filtration and washing with ethanol (3 x 0.5 mL) of the crude reaction mixture and column chromatography (toluene) as a yellow solid (57.9 mg, 93 %).

R_F = 0.28 (toluene).

^{1}H NMR (600 MHz, $CDCl_3$): δ 8.75 – 8.70 (m, 1H), 8.69 – 8.61 (m, 1H), 8.40 (dd, *J* = 14.7, 1.0 Hz, 1H), 8.26 – 8.19 (m, 1H), 7.95 – 7.89 (m, 4H), 7.78 (dd, *J* = 5.4, 3.0 Hz, 2H), 7.73 – 7.57 (m, 4H), 7.44 (d, *J* = 14.7 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$): δ 166.5, 134.6, 132.5, 131.8, 131.7, 130.7, 130.4, 130.3, 128.7, 126.8, 126.8, 126.7, 126.6, 124.8, 124.2, 123.7, 123.0, 122.5, 119.6, 118.2.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for $C_{24}H_{15}NNaO_2^+$: 372.0995, found: 372.0995.

FT-IR (neat, ν_{max} cm^{-1}): 3105, 3057, 1768, 1711, 981.

Preparation of triarylboroxines from arylboronic acids

Triarylboroxines (**6a-6f**) were prepared according to two procedures, which were modified from literature[14]

General procedure for a "small scale" reaction

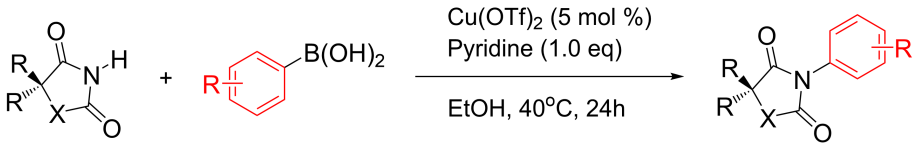
A round-bottom flask was charged with arylboronic acid (0.44 mmol) and dry toluene (5 mL), heated to 130 °C and allowed to stir for 1-3 hours, after which the solvent was removed under reduced pressure. The resulting triarylboroxine was used in the following *N*-arylation reaction without further purification.

General procedure for "large scale" reaction

A round-bottom flask was charged with arylboronic acid (1.00 g, 4.27 - 8.20 mmol) and dry toluene (20 mL) before being connected to a vacuum distillation setup and evacuated to ca. 85 mmHg. The flask was heated to 65 – 70 °C and the toluene was distilled off until near completion. The flask was then charged with fresh toluene and the process was repeated twice more. Remaining solvent was evaporated at 70 °C on a rotary evaporator at a reduced pressure. A portion (0.44 mmol) of resulting triarylboroxine was used in the following *N*-arylation reaction without further purification. The remaining triarylboroxine was stored under dry atmosphere.

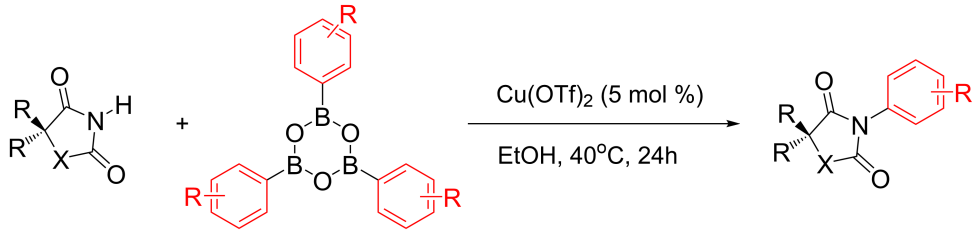
Synthesis of *N*-arylhydantoins

Method 1 (arylboronic acids) – general procedure



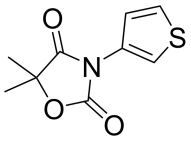
Cyclic imide (0.40 mmol, 1.0 eq), arylboronic acid (1.2 mmol, 3.0 eq), $Cu(OTf)_2$ (7.2 mg, 0.020 mmol, 0.050 eq) and ethanol (2 mL) were added to a 5 mL round-bottom flask equipped with a magnetic stir bar. Pyridine (32 µL, 0.40 mmol, 1.0 eq) was added via a syringe, a cooler was connected to the flask and the mixture was stirred with access to air at 40°C for 24 hours. The resulting mixture was allowed to cool to room temperature before it was concentrated under reduced pressure then purified by column chromatography using silica gel and eluent system as specified.

Method 2 (arylboroxines) – general procedure



Cyclic imide (0.40 mmol, 1.0 eq), arylboroxine (0.44 mmol, 1.1 eq), $Cu(OTf)_2$ (7.2 mg, 0.020 mmol, 0.050 eq) and ethanol (2 mL) were added to a 5 mL round-bottom flask equipped with a magnetic stir bar. A cooler was connected to the flask and the mixture was stirred with access to air at 40°C for 24 hours. The resulting mixture was allowed to cool to room temperature before it was concentrated under reduced pressure then purified by column chromatography using silica gel and eluent system as specified.

5,5-Dimethyl-3-(3-thiophenyl)-2,4-oxazolidinedione (7a) [NEW]



Following the general procedure for **method 1** at a 0.14 mmol scale using 1 mL of ethanol, **7a** was obtained after column chromatography (chloroform:hexane:acetone [1:8:1]) as slightly yellow solid (29.5 mg, 100 %).

R_F = 0.31 (chloroform:hexane:acetone [1:8:1]).

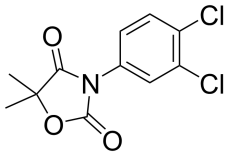
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.76 (dd, *J* = 3.3, 1.4, 1H), 7.58 (dd, *J* = 5.3, 1.4, 1H), 7.38 (dd, *J* = 5.3, 3.3, 1H), 1.66 (s, 6H).

^{13}C NMR (150 MHz, $CDCl_3$): δ (ppm): 174.0, 152.5, 129.0, 125.2, 122.0, 117.4, 83.0, 23.8.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for $C_9H_9NNaO_3S^+$: 234.0195, found: 234.0195.

FT-IR (neat, ν_{max} cm^{-1}): 3106, 2940, 1815, 1726, 1437, 629.

5,5-Dimethyl-3-(3,4-dichlorophenyl)-2,4-oxazolidinedione (7b) [CAS: 35992-67-7]



Following the general procedure for **method 1** at a 0.14 mmol scale using 1 mL of ethanol, **7b** was obtained after column chromatography (chloroform:hexane:acetone [1:8:1→ 1:5:1]) as colorless solid (38.0 mg, 98 %).

R_F = 0.40 (chloroform:hexane:acetone [1:5:1]).

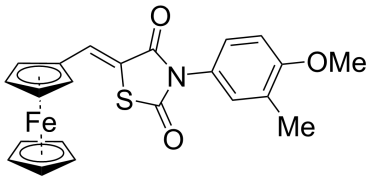
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.66 (d, *J* = 2.5 Hz, 1H), 7.57 (d, *J* = 8.7 Hz, 1H), 7.39 (dd, *J* = 8.7, 2.5 Hz, 1H), 1.69 (s, 6H).

^{13}C NMR (151 MHz, $CDCl_3$): δ 174.2, 152.4, 133.3, 133.0, 130.9, 130.2, 127.1, 124.4, 83.6, 23.8.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for $C_{11}H_9{}^{35}Cl_2NNaO_3^+$: 295.9852, found: 295.9851.

This compound has been reported in the literature.[15]

(*Z*)-5-ferrocenylidene-3-(4-methoxy-3-methylphenyl)-2,4-thiazolidinedione (7e) [NEW]



Following the general procedure for **method 1**, **7e** was obtained after column chromatography (toluene:acetone [70:1]) as a wine-red solid (167.3 mg, 97 %).

R_F = 0.55 (toluene:acetone [70:1], red spot).

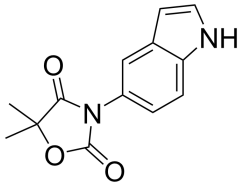
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.84 (s, 1H), 7.13 (dd, *J* = 8.6, 2.7 Hz, 1H), 7.11 – 7.09 (m, 1H), 6.93 (d, *J* = 8.6 Hz, 1H), 4.60 (dt, *J* = 19.2, 1.9 Hz, 4H), 4.25 (s, 5H), 3.87 (s, 3H), 2.25 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$): δ 167.8, 165.5, 158.2, 136.7, 129.3, 128.1, 125.9, 124.9, 116.8, 110.3, 76.6, 72.2, 70.7, 70.0, 55.5, 16.3.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for $C_{22}H_{19}FeNNaO_3S^+$: 456.0326, found: 456.0327.

FT-IR (neat, ν_{max} cm^{-1}): 3094, 3021, 2942, 1734, 1677, 1600, 1362, 1246, 818.

5,5-Dimethyl-(1*H*-5-indolyl)-2,4-oxazolidinedione (7f) [NEW]



Following the general procedure for **method 1** at a 0.14 mmol scale using 1 mL of ethanol, **7f** was obtained after column chromatography. Three consecutive columns were necessary for the purification of **7f**: chloroform:hexane:acetone [1:5:1], then hexane:acetone [3:2], then Et_2O:hexane [7:3]. The desired fractions were collected and the solvent was removed after each column before the mixture of product and impurities was re-applied on to the next column. **7f** was obtained as a brown solid (25.5 mg, 75 %).

R_F = 0.28 (Et_2O).

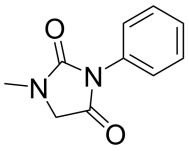
^{1}H NMR (600 MHz, $CDCl_3$): δ 8.35 (br s, 1H), 7.64 (d, *J* = 2.0, 1H), 7.44 (d, *J* = 8.6, 1H), 7.28-7.26 (m, 1H), 7.13 (dd, *J* = 8.6, 2.0, 1H), 6.60-6.58 (m, 1H), 1.71 (s, 6H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 175.6, 154.2, 135.5, 128.1, 125.8, 123.0, 119.7, 118.8, 111.7, 103.4, 83.4, 23.8.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for $C_{13}H_{12}N_2NaO_3^+$: 267.0740, found: 267.0740.

FT-IR (neat, V_{max} cm^{-1}): 3315, 3097, 2937, 1820, 1716, 1456.

1-Methyl-3-phenylhydantoin (7g) [CAS: 2221-12-7]



Following the general procedure for **method 2**, **7g** was obtained after column chromatography (chloroform:hexane:acetone [7:2:1]) as a colorless solid (63.7 mg, 83 %).

R_F = 0.27 (chloroform:hexane:acetone [7:2:1]).

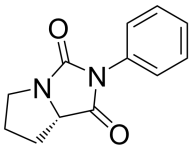
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.48 – 7.43 (m, 2H), 7.41 – 7.38 (m, 2H), 7.36 (td, *J* = 7.2, 1.3 Hz, 1H), 4.02 (s, 2H), 3.07 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$): δ 168.6, 155.7, 131.8, 129.0, 128.1, 126.0, 51.6, 29.8.

HRMS (ESI) m/z $[M + Na]^+$: Calcd. for $C_{10}H_{10}N_2NaO_2^+$: 213.0634, found: 213.0634.

The spectroscopic data is in accordance with the literature.[16]

(*S*)-2-phenyltetrahydro-1H-pyrrolo(1,2-c)imidazole-1,3(2H)-dione (7h) [CAS: 70741-88-7]



Following the general procedure for **method 2**, **7h** was obtained after column chromatography (chloroform:hexane:acetone [3:6:1]) as a colorless solid (74.2 mg, 86 %).

R_F = 0.23 (chloroform:hexane:acetone [3:6:1]).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.48 – 7.43 (m, 2H), 7.41 – 7.37 (m, 2H), 7.37 – 7.33 (m, 1H), 4.23 (dd, *J* = 9.3, 7.5 Hz, 1H), 3.79 (dt, *J* = 11.3, 7.7 Hz, 1H), 3.33 (ddd, *J* = 11.3, 8.4, 4.5 Hz, 1H), 2.34 (dtd, *J* = 12.7, 7.3, 3.6 Hz, 1H), 2.21 – 2.13 (m, 1H), 2.14 – 2.05 (m, 1H), 1.91 – 1.79 (m, 1H).

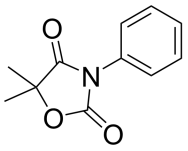
^{13}C NMR (151 MHz, $CDCl_3$): δ 172.5, 159.3, 131.8, 129.0, 128.1, 125.8, 63.2, 45.7, 27.7, 26.8.

HRMS (ESI) m/z $[M + Na]^+$: Calcd. for $C_{12}H_{12}N_2NaO_2^+$: 239.0791, found: 239.0790.

$(\alpha)_D^{20}$ = - 44.4 (c = 0.45, $CHCl_3$).

The spectroscopic data is in accordance with the literature.[17]

5,5-Dimethyl-3-phenyl-2,4-oxazolidinedione (7i) [CAS: 24201-26-1]



Following the general procedure for **method 2**, **7i** was obtained after column chromatography (chloroform:hexane:acetone [1:8:1]) as a colorless solid (77.7 mg, 95 %).

R_F = 0.24 (chloroform:hexane:acetone [1:8:1]).

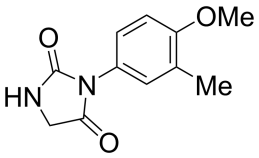
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.52 – 7.47 (m, 2H), 7.46 – 7.39 (m, 3H), 1.69 (s, 6H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 174.9, 153.3, 130.9, 129.3, 128.8, 125.5, 83.4, 23.8.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for $C_{11}H_{11}NNaO_3^+$: 228.0631, found: 228.0631.

The spectroscopic data is in accordance with the literature.[18]

3-(4-methoxy-3-methylphenyl)hydantoin (7j) [CAS: 1693615-54-1]



Following the general procedure for **method 2** at a 0.14 mmol scale using 1 mL of ethanol, **7j** was obtained after column chromatography (hexane:acetone [3:1 → 1:1]) as an off-white solid (22.5 mg, 73 %).

R_F = 0.27 (hexane:acetone [1:1]).

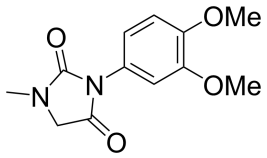
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.15 (ddd, *J* = 8.6, 2.6, 0.7 Hz, 1H), 7.13 – 7.11 (m, 1H), 6.90 (d, *J* = 8.6 Hz, 1H), 6.04 (br s, 1H), 4.11 (d, *J* = 1.2 Hz, 2H), 3.85 (s, 3H), 2.24 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$): δ 170.4, 157.8, 157.7, 128.6, 127.9, 125.1, 123.2, 110.2, 55.5, 46.4, 16.3.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for $C_{11}H_{12}N_2NaO_3^+$: 243.0740, found: 243.0739.

This compound has been reported in the literature, but without spectroscopic data.

3-(3,4-dimethoxyphenyl)-1-methylhydantoin (7k) [NEW]



Following the general procedure for **method 2**, **7k** was obtained after column chromatography (chloroform:hexane:acetone [7:2:1]) as a colorless solid (80.4 mg, 81 %).

R_F = 0.20 (chloroform:hexane:acetone [7:2:1]).

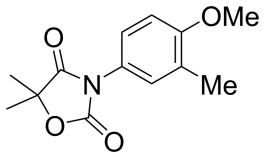
^{1}H NMR (600 MHz, $CDCl_3$): δ 6.94 (dd, *J* = 8.6, 2.1 Hz, 1H), 6.92 (d, *J* = 8.5 Hz, 1H), 6.88 (d, *J* = 2.1 Hz, 1H), 4.02 (s, 2H), 3.89 (s, 3H), 3.87 (s, 3H), 3.07 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$): δ 168.9, 156.0, 149.2, 148.9, 124.5, 118.8, 111.1, 109.8, 56.02, 55.96, 51.6, 29.9.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for $C_{12}H_{14}N_2NaO_4^+$: 273.0846, found: 273.0845.

FT-IR (neat, ν_{max} cm^{-1}): 3096, 2924, 1761, 1706, 1447, 1232.

5,5-Dimethyl-3-(4-methoxy-3-methylphenyl)-2,4-oxazolidinedione (7l) [NEW]



Following the general procedure for **method 2** at a 0.14 mmol scale using 1 mL of ethanol, **7l** was obtained after column chromatography (chloroform:hexane:acetone [1:8:1 → 1:5:1]) as pale yellow solid (33.2 mg, 96 %).

R_F = 0.43 (chloroform:hexane:acetone [1:8:1]).

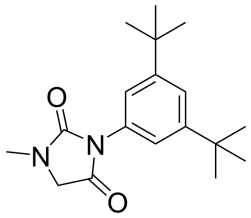
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.17 (dd, *J* = 8.6, 2.6, 1H), 7.14 (d, *J* = 2.5, 1H), 6.89 (d, *J* = 8.6, 1H), 3.85, (s, 3H), 2.24 (s, 3H), 1.67 (s, 6H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 175.2, 158.0, 153.7, 128.0, 127.9, 124.4, 122.9, 110.1, 83.3, 55.5, 23.7, 16.2.

HRMS (ESI) m/z [M + Na]$^+$: Calcd. for $C_{13}H_{15}NNaO_4^+$: 272.0893, found: 272.0893.

FT-IR (neat, V_{max} cm^{-1}): 3029, 2966, 2941, 2918, 1815, 1720, 1431, 1350.

3-(3,5-di-*tert*-butylphenyl)-1-methylhydantoin (7m) [NEW]



Following the general procedure for **method 2, 7m** was obtained after column chromatography (chloroform:hexane:acetone [5:4:1]) as a colorless, sticky oil (112.0 mg, 93 %).

R_F = 0.35 (chloroform:hexane:acetone [5:4:1]).

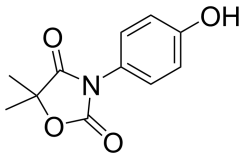
^{1}H NMR (600 MHz, $CDCl_3$): δ 7.42 (t, *J* = 1.8 Hz, 1H), 7.15 (d, *J* = 1.8 Hz, 2H), 4.03 (s, 2H), 3.09 (s, 3H), 1.32 (s, 18H).

^{13}C NMR (151 MHz, $CDCl_3$): δ 168.9, 156.2, 151.6, 131.0, 122.6, 120.7, 51.7, 34.9, 31.3, 29.8.

HRMS (ESI) m/z $[M + Na]^+$: Calcd. for $C_{12}H_{14}N_2NaO_4^+$: 325.1886, found: 325.1886.

FT-IR (neat, ν_{max} cm^{-1}): 2960, 1779, 1715, 1447, 1438.

5,5-Dimethyl-3-(4-hydroxyphenyl)-2,4-oxazolidinedione (7n) [NEW]



Following the general procedure for **method 1** at a 0.14 mmol scale using 1 mL of ethanol, **7n** obtained after column chromatography (chloroform:hexane:acetone [1:3:1]) as colorless solid (23.1 mg, 75 %).

R_F = 0.20 (chloroform:hexane:acetone [1:3:1]).

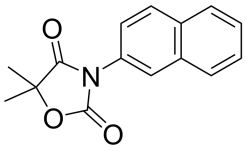
^{1}H NMR (600 MHz, Acetone-d_6): δ 8.74 (br s, 1H), 7.29 – 7.25 (m, 2H), 6.95 – 6.92 (m, 2H), 1.65 (s, 6H).

^{13}C NMR (151 MHz, Acetone-d_6): δ 175.9, 158.5, 154.4, 128.8, 124.3, 116.3, 83.9, 23.7.

HRMS (ESI) m/z $[M + Na]^+$: Calcd. for $C_{11}H_{11}NNaO_4^+$: 244.0580, found: 244.0580.

FT-IR (neat, V_{max} cm^{-1}): 3374, 3351, 2987, 1806, 1710, 1450, 1368.

5,5-Dimethyl-3-(2-naphthalenyl)-2,4-oxazolidinedione (7o) [NEW]



Following the general procedure for **method 2** at a 0.14 mmol scale using 1.5 mL of ethanol, **7o** was obtained after column chromatography (chloroform:hexane:acetone [1:5:1]) as pale yellow solid (35.6 mg, 83 %).

R_F = 0.36 (chloroform:hexane:acetone [1:5:1]).

^{1}H NMR (600 MHz, $CDCl_3$): δ 7.97-7.93 (m, 2H), 7.90-7.86 (m, 2H), 7.58-7.51 (m, 3H), 1.73 (s, 6H).

^{13}C NMR (150 MHz, $CDCl_3$): δ 175.0, 153.4, 133.0, 132.8, 129.3, 128.3, 128.2, 127.7, 127.1, 126.8, 124.7, 122.7, 83.5, 23.8.

FT-IR (neat, ν_{max} cm^{-1}): 3058, 2923, 1815, 1725, 1414, 744.

HRMS (ESI) m/z $[M + Na]^+$: Calcd. for $C_{15}H_{13}NNaO_3^+$: 278.0788, found: 278.0787.

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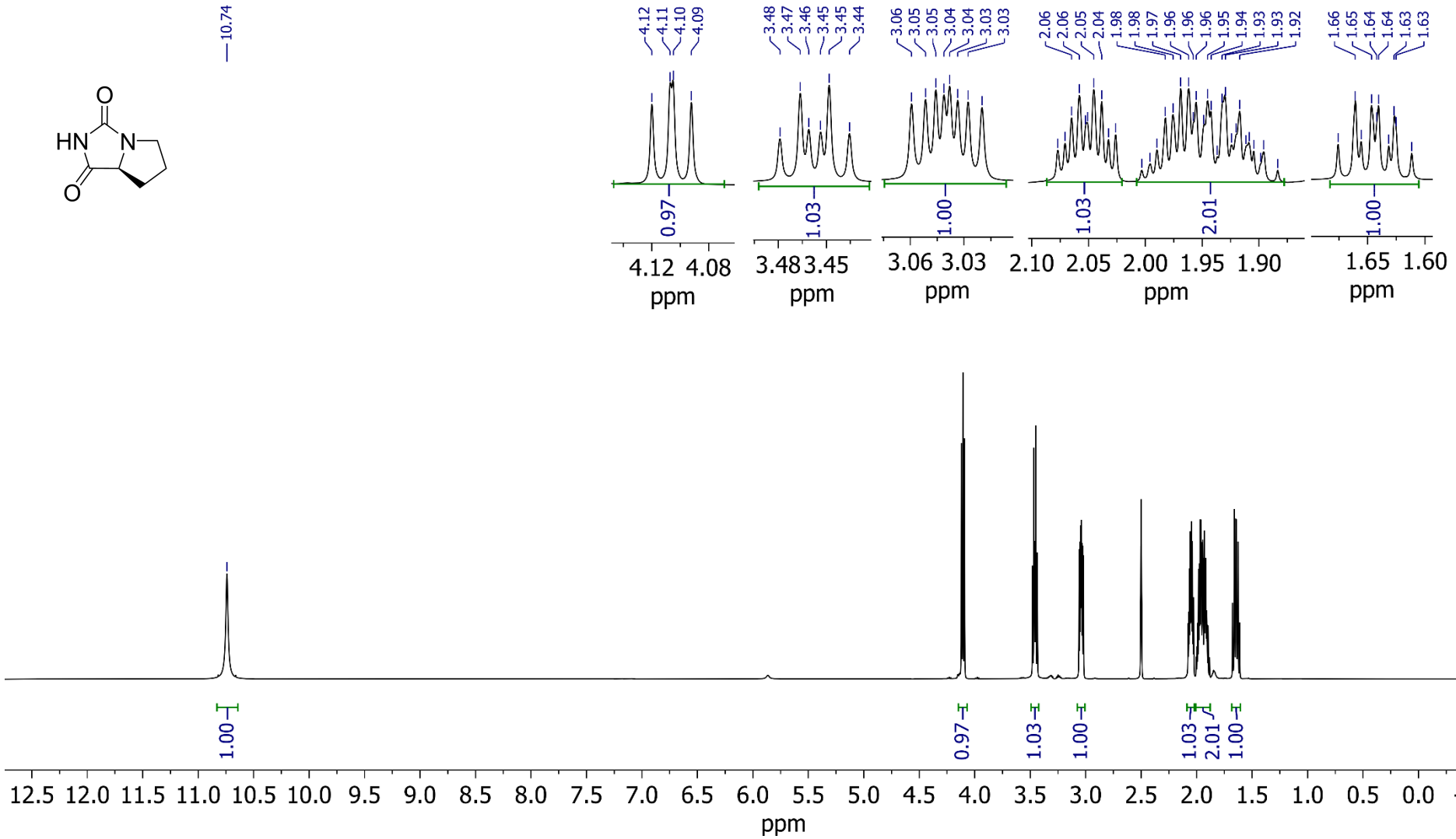
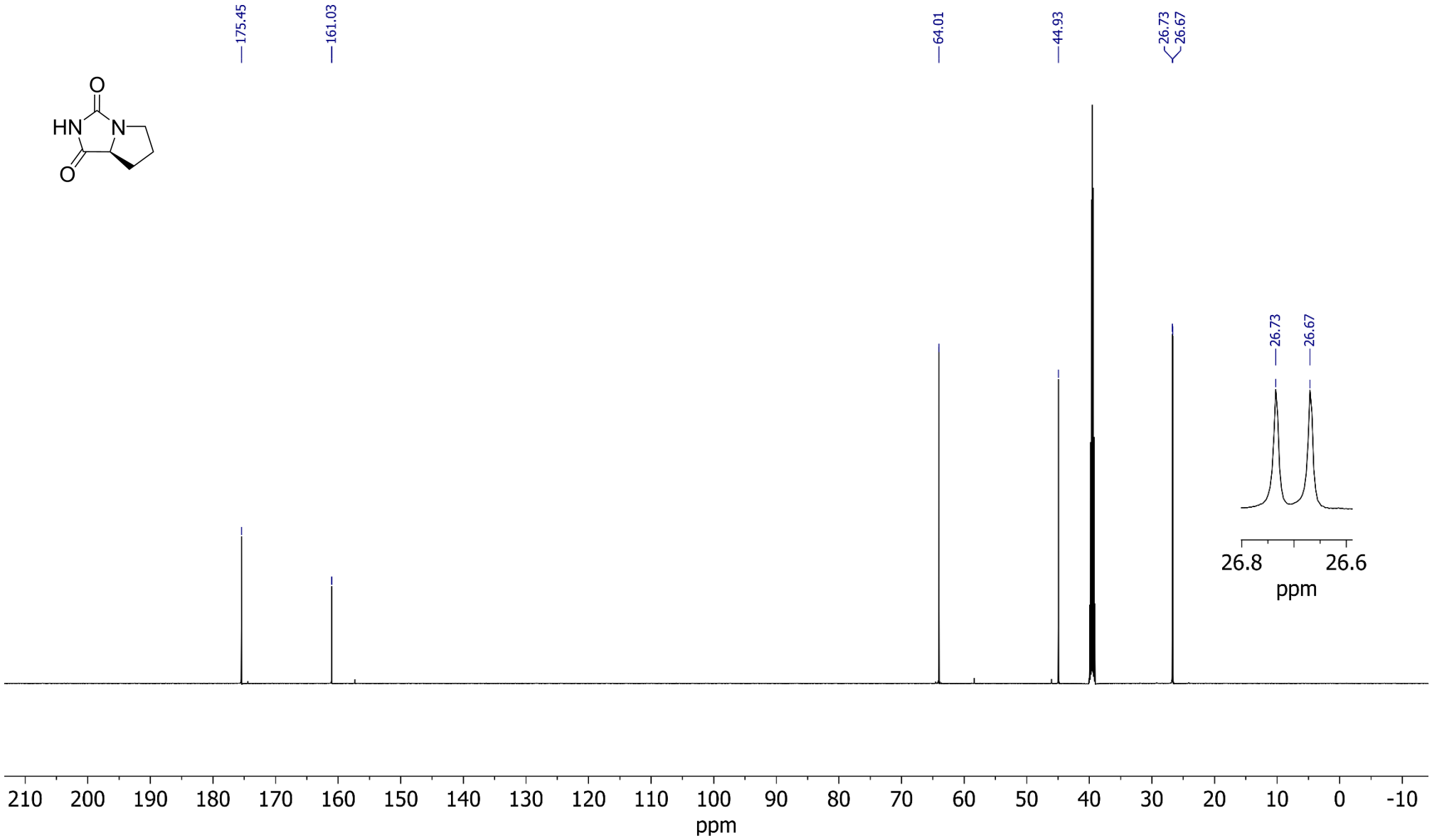
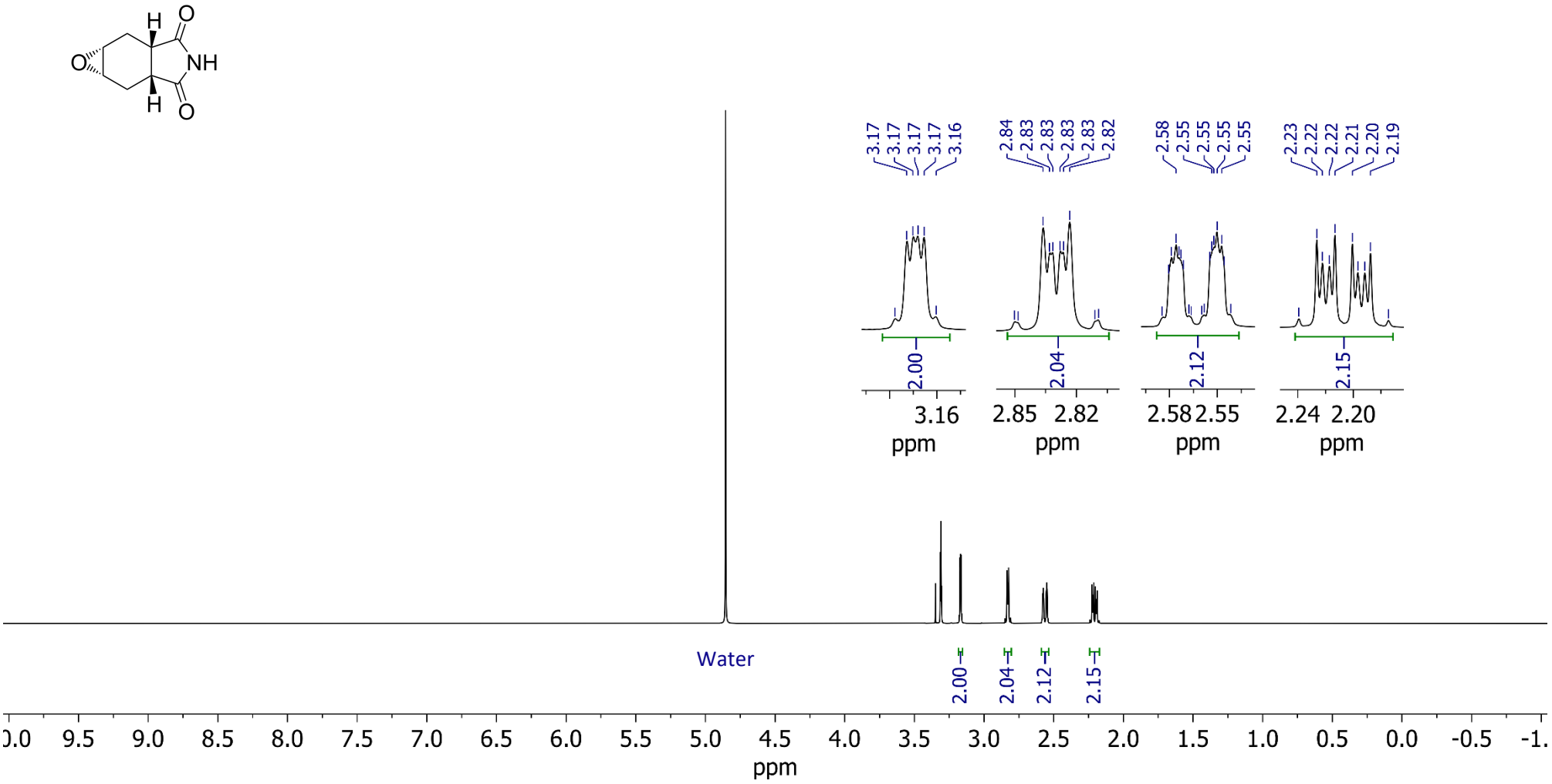
^{1}H NMR spectrum of 1b (600 MHz, DMSO-d_6)



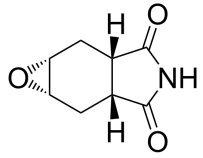
^{13}C NMR spectrum of 1b (151 MHz, DMSO-d_6)



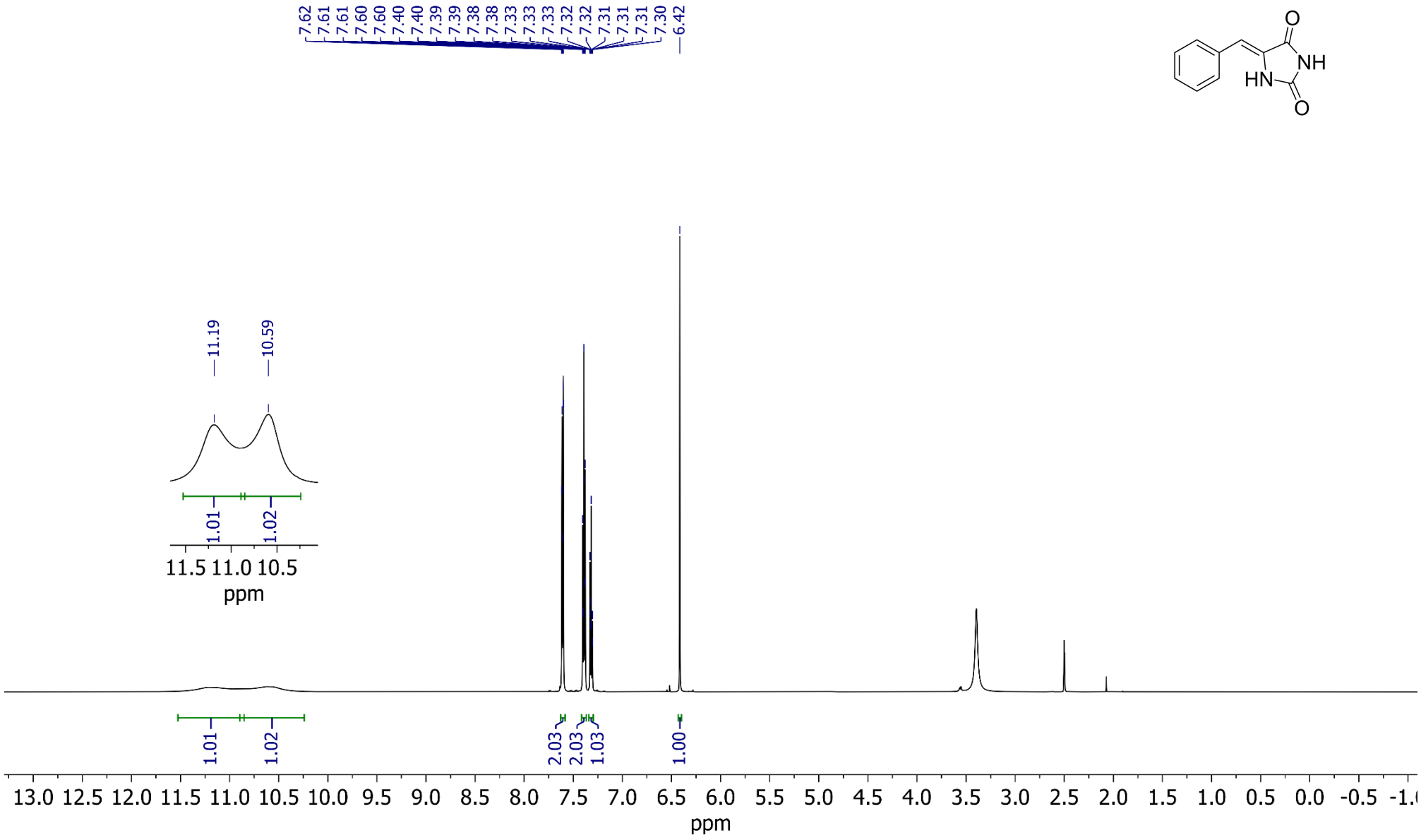
^{1}H NMR spectrum of 1k (600 MHz, CD_3OD)



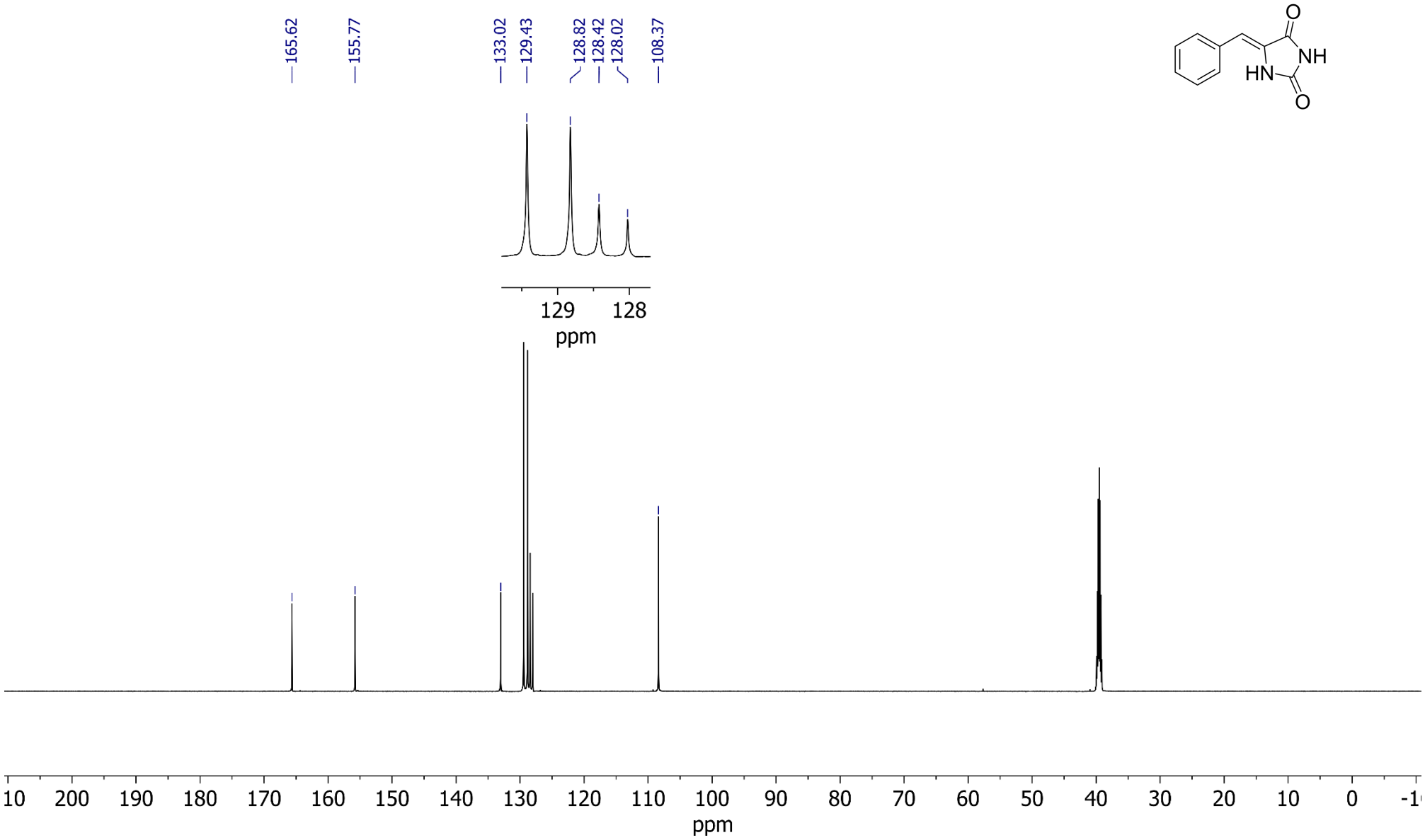
^{13}C NMR spectrum of 1k (151 MHz, CD_3OD)



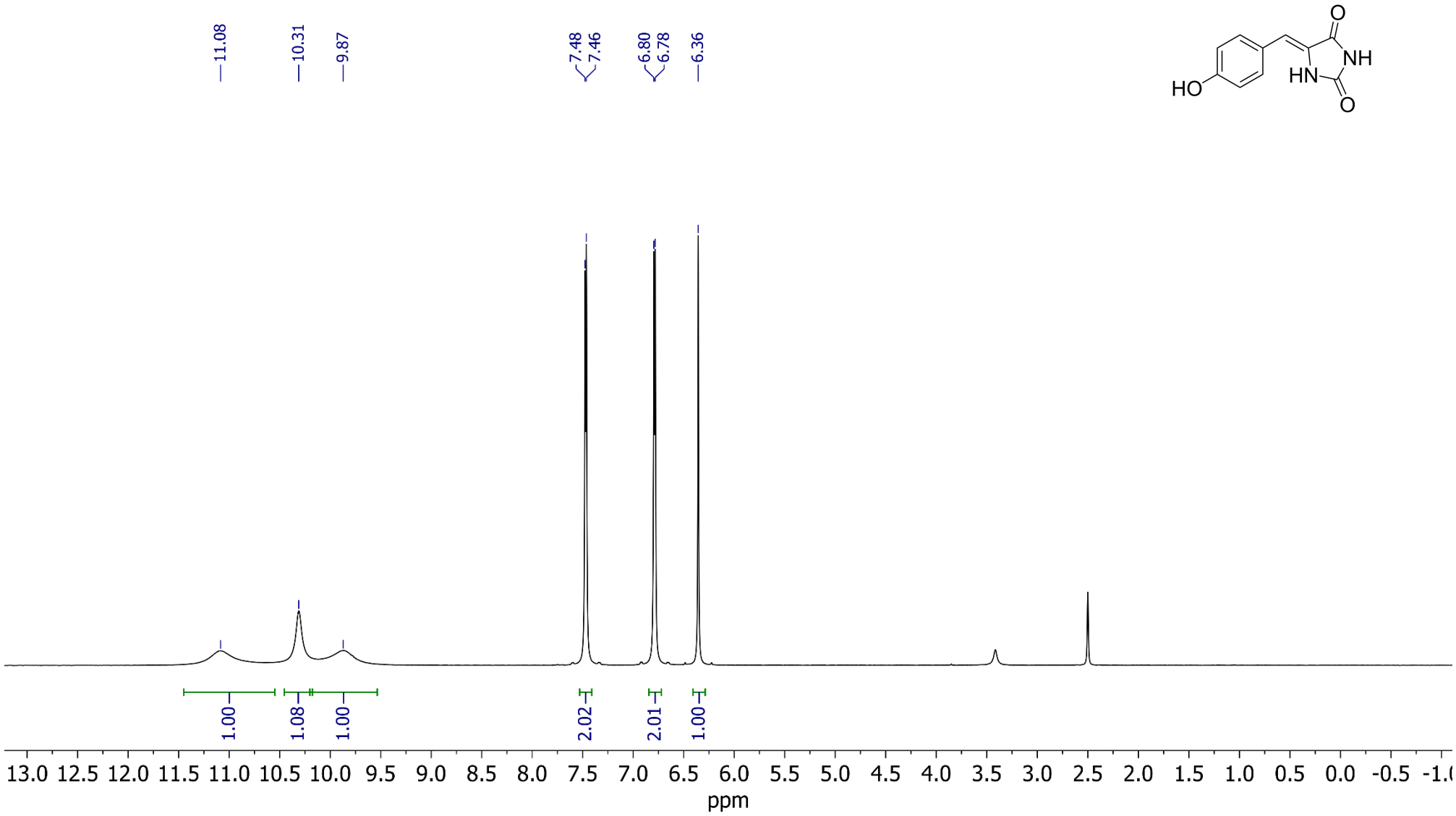
^{1}H NMR spectrum of 1g (600 MHz, DMSO-d_6)



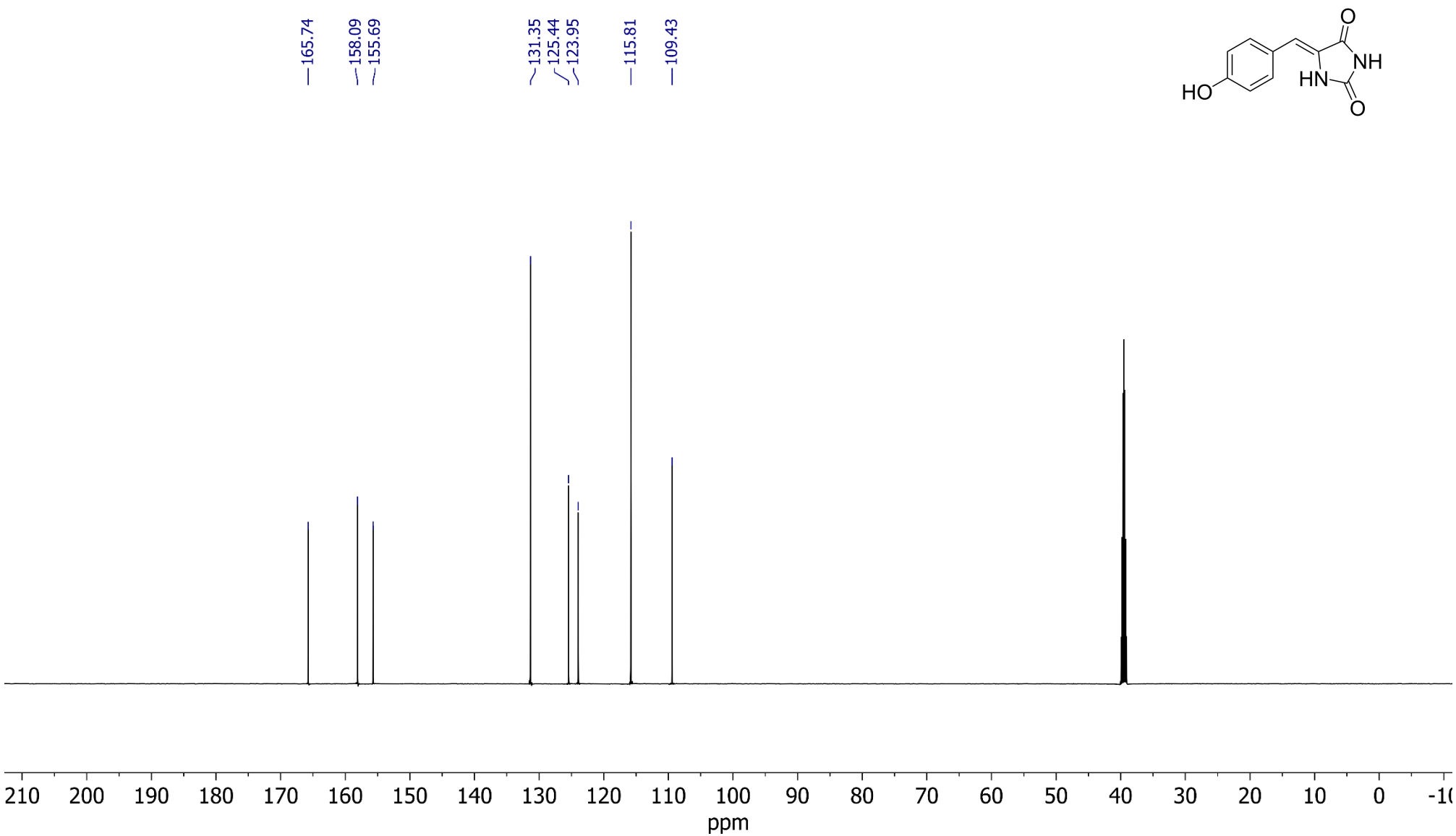
^{13}C NMR spectrum of 1g (151 MHz, DMSO-d_6)



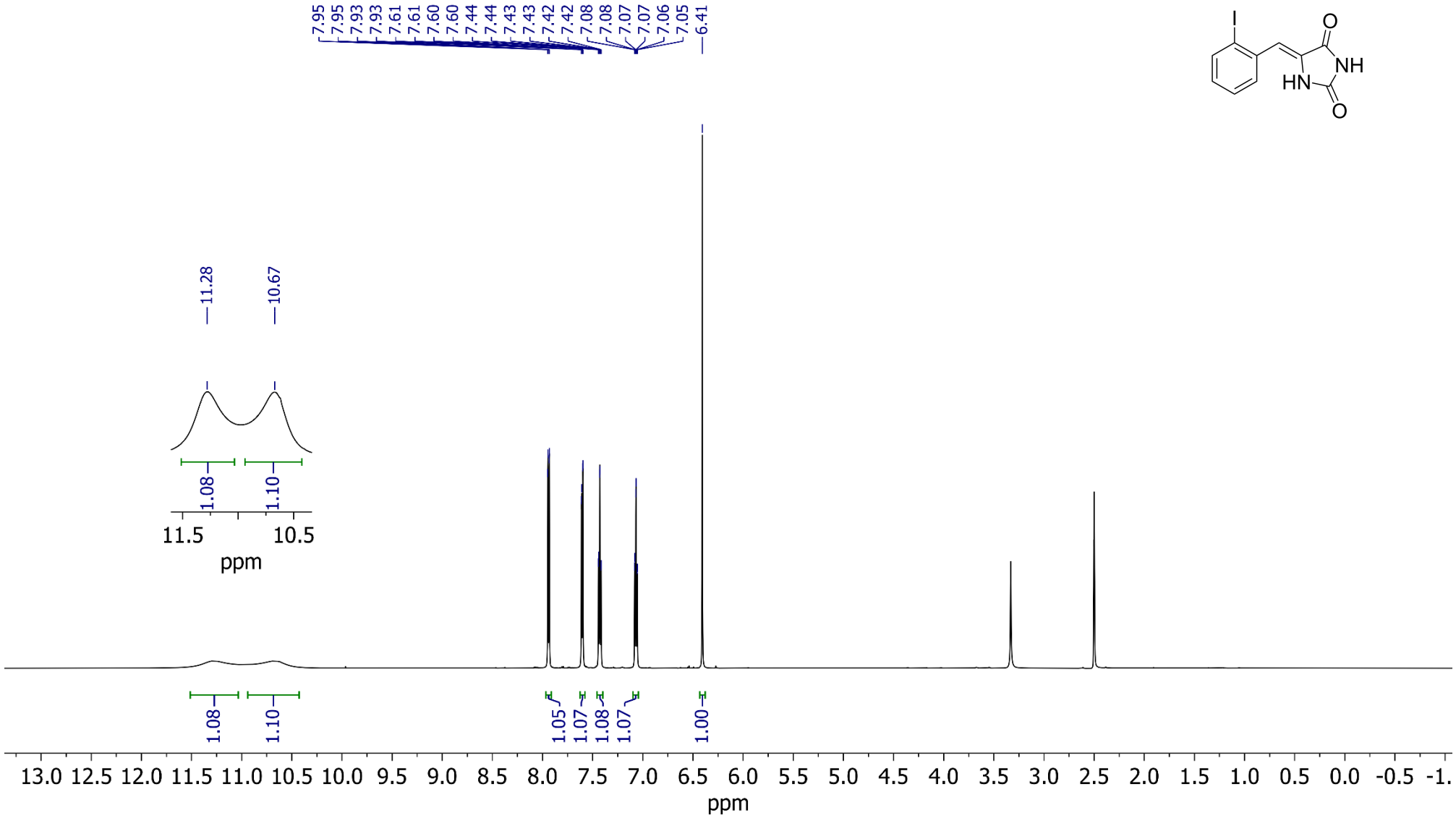
^{1}H NMR spectrum of 1h (600 MHz, DMSO-d_6)



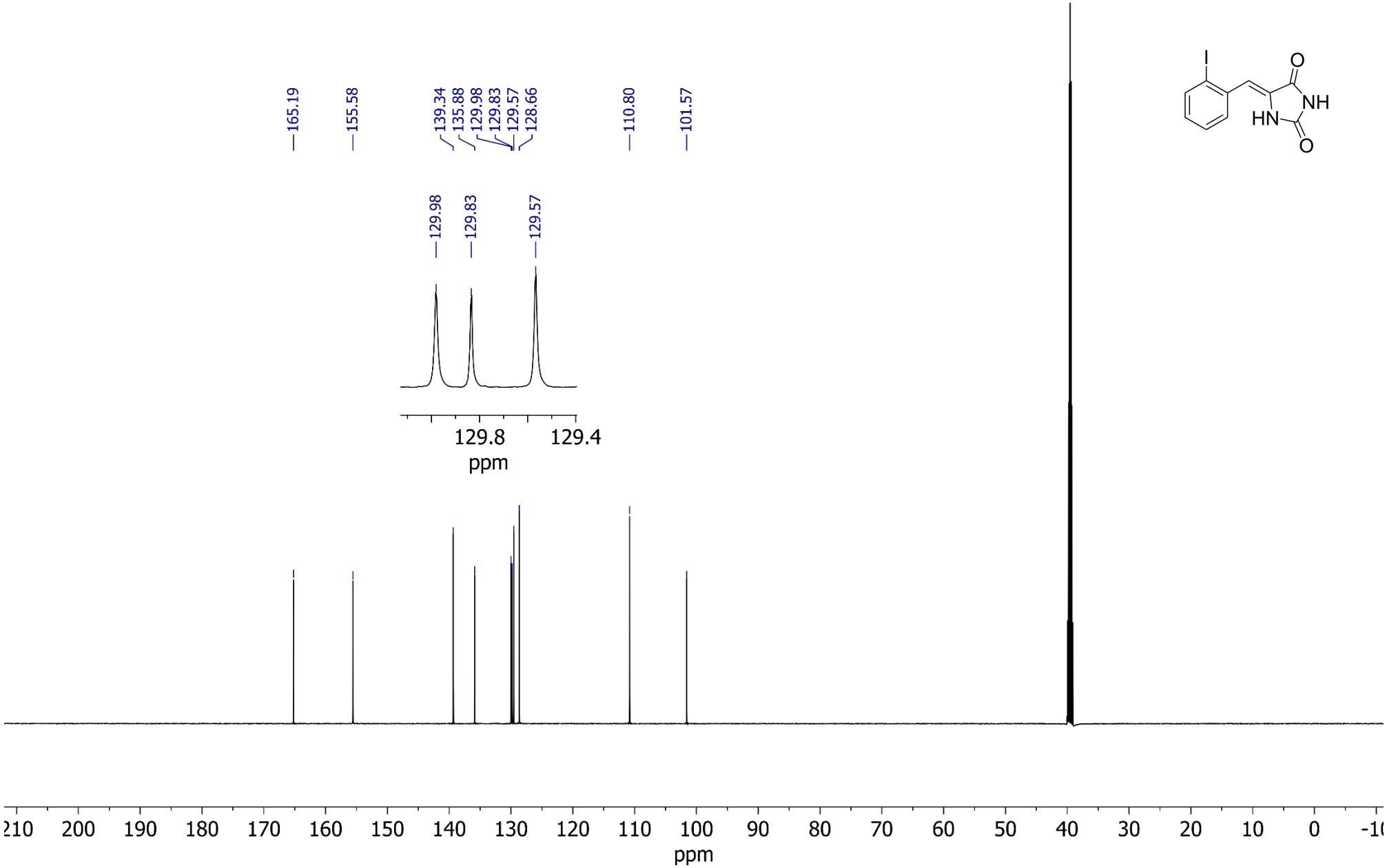
^{13}C NMR spectrum of 1h (151 MHz, DMSO-d_6)



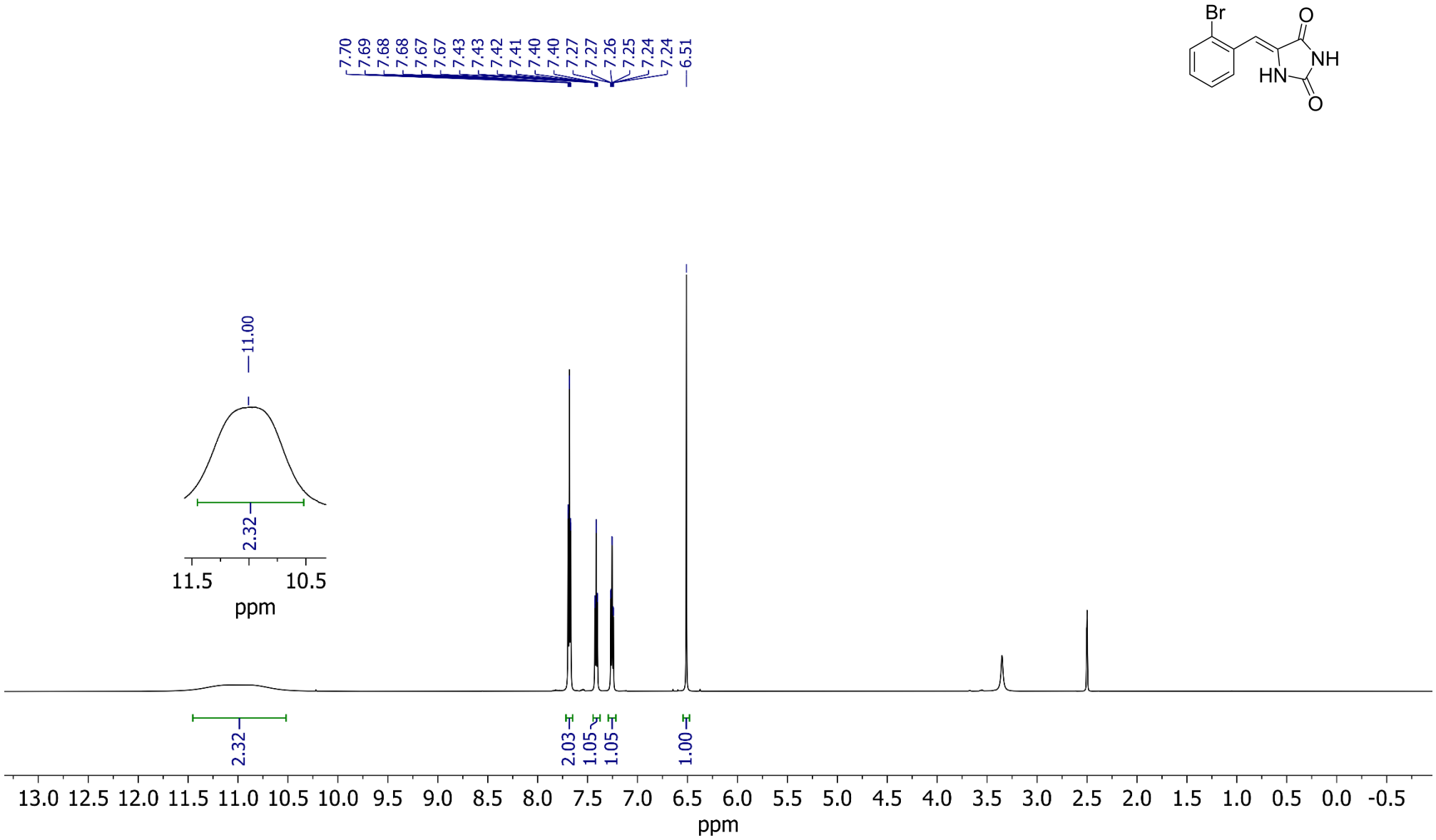
^{1}H NMR spectrum of 1i (600 MHz, DMSO-d_6)



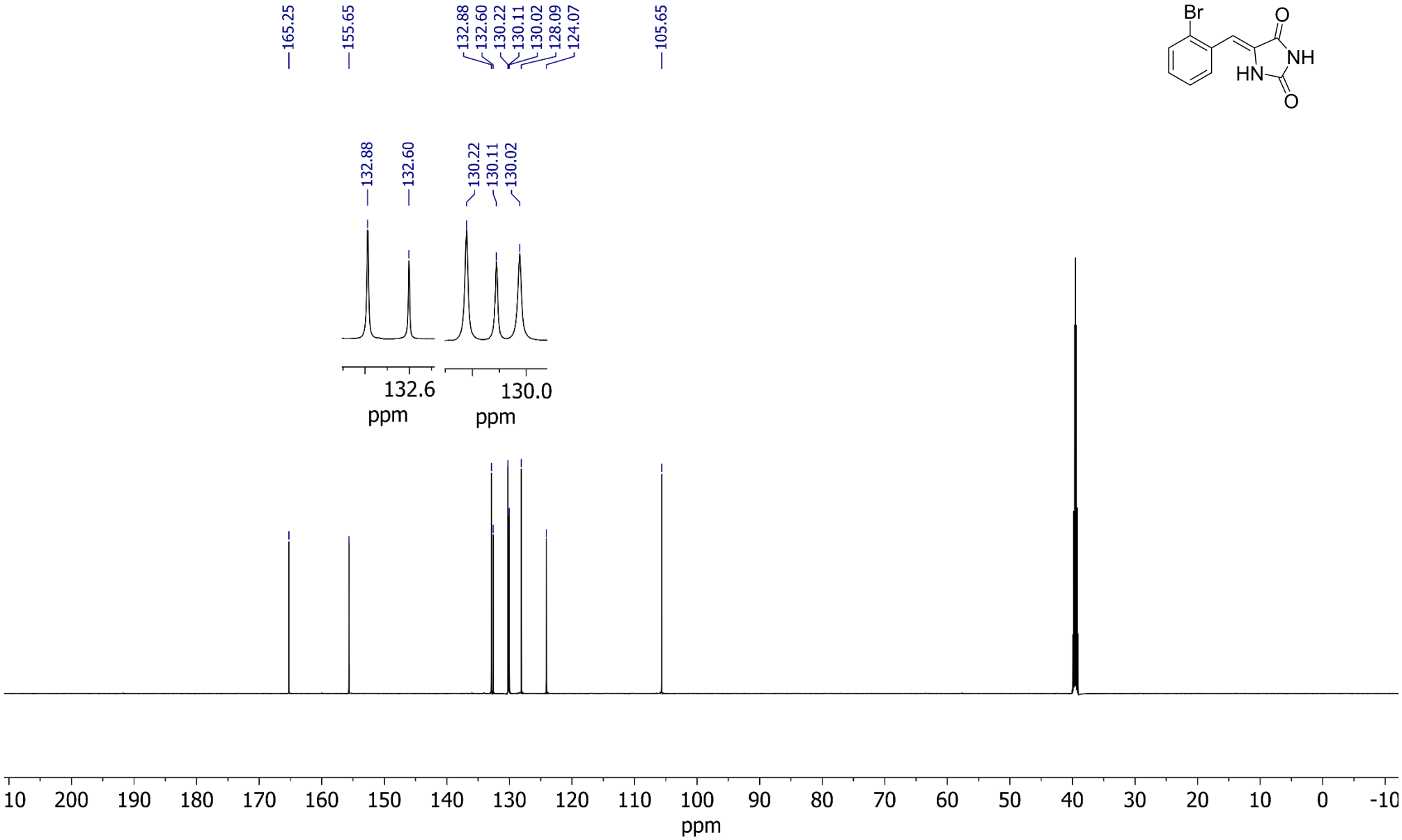
^{13}C NMR spectrum of 1i (151 MHz, DMSO-d_6)



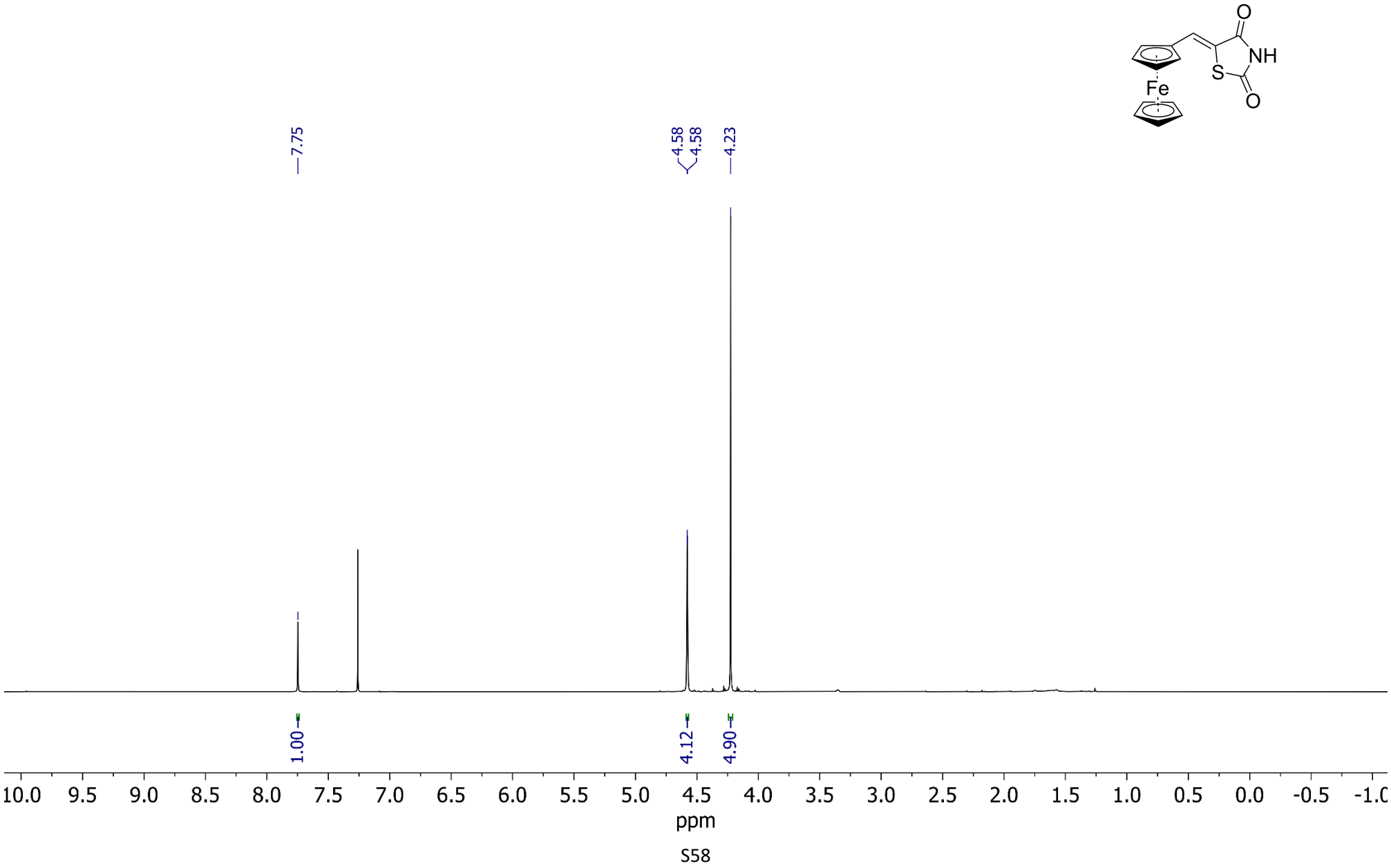
^{1}H NMR spectrum of 1j (600 MHz, DMSO-d_6)



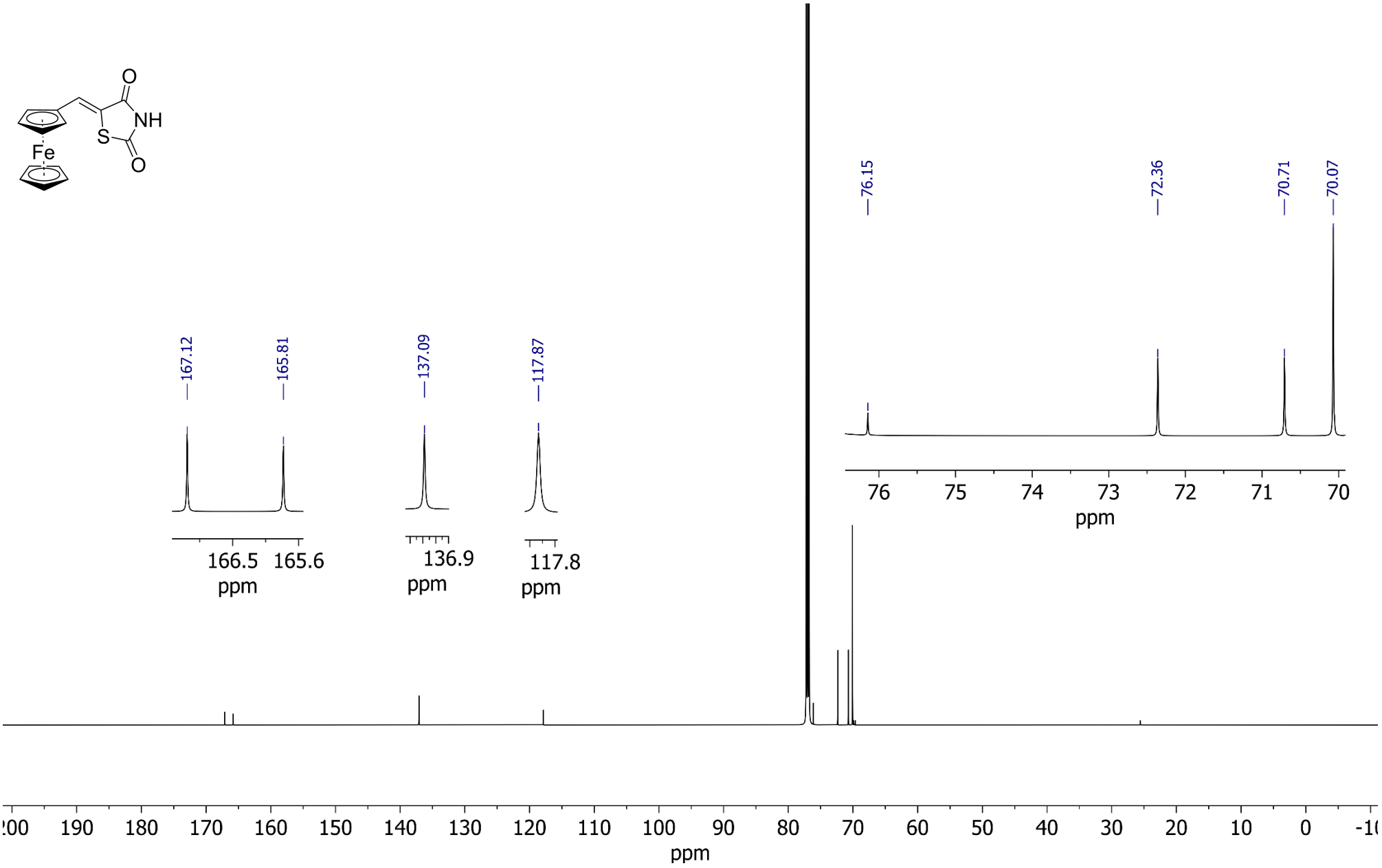
^{13}C NMR spectrum of 1j (151 MHz, DMSO-d_6)



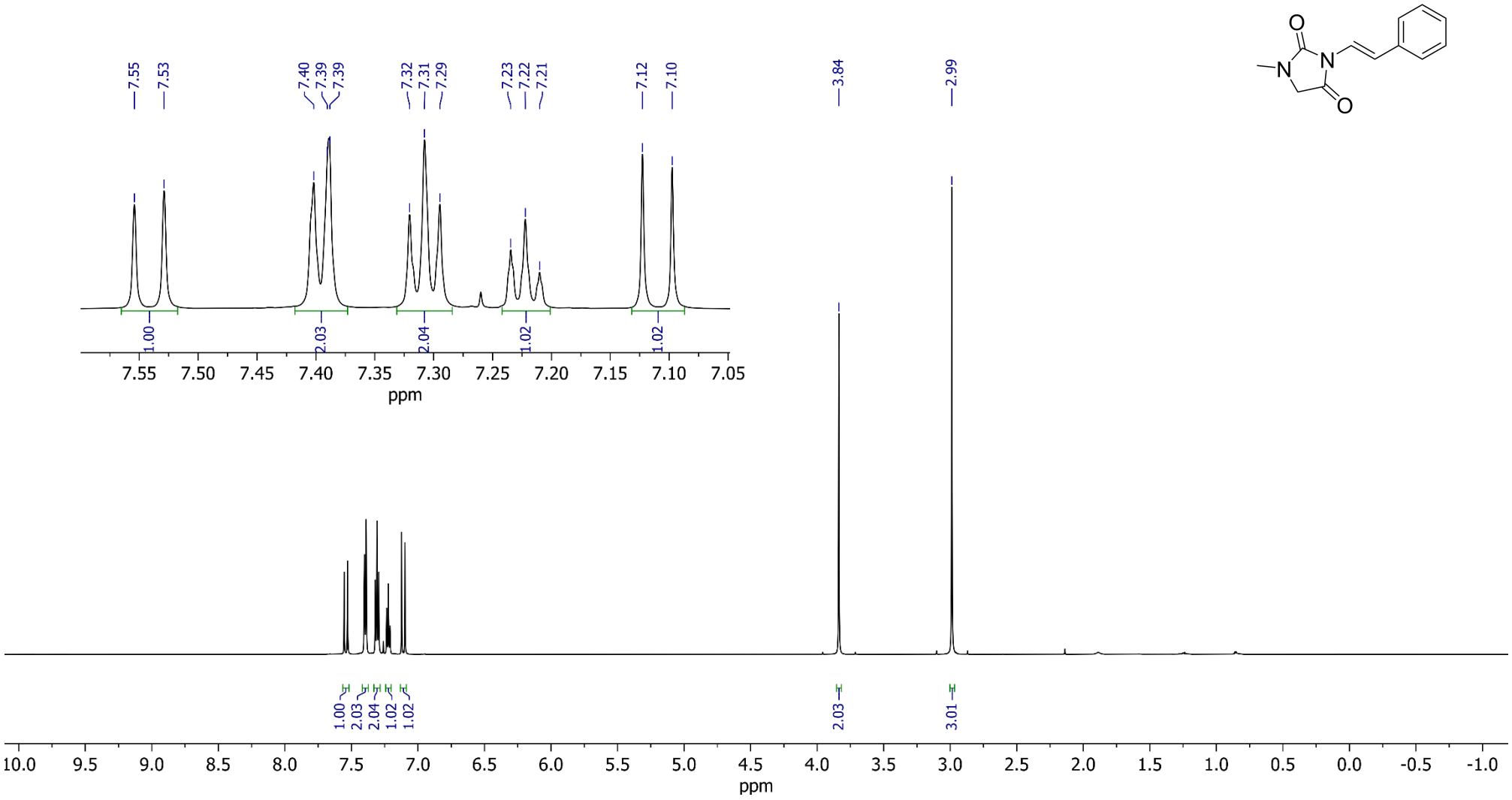
^{1}H NMR spectrum of 1f (600 MHz, $CDCl_3$)



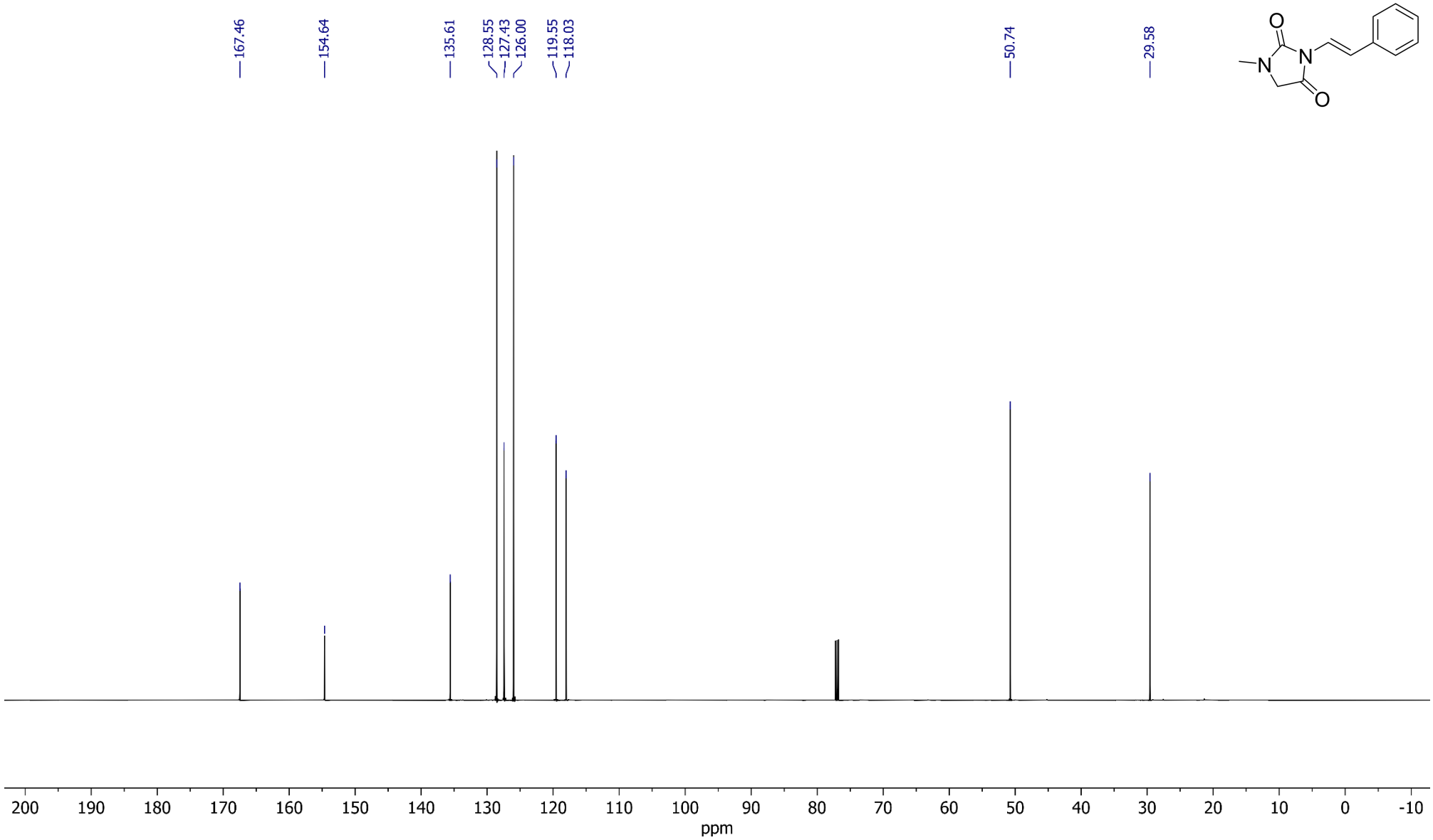
^{13}C NMR spectrum of 1f (151 MHz, $CDCl_3$)



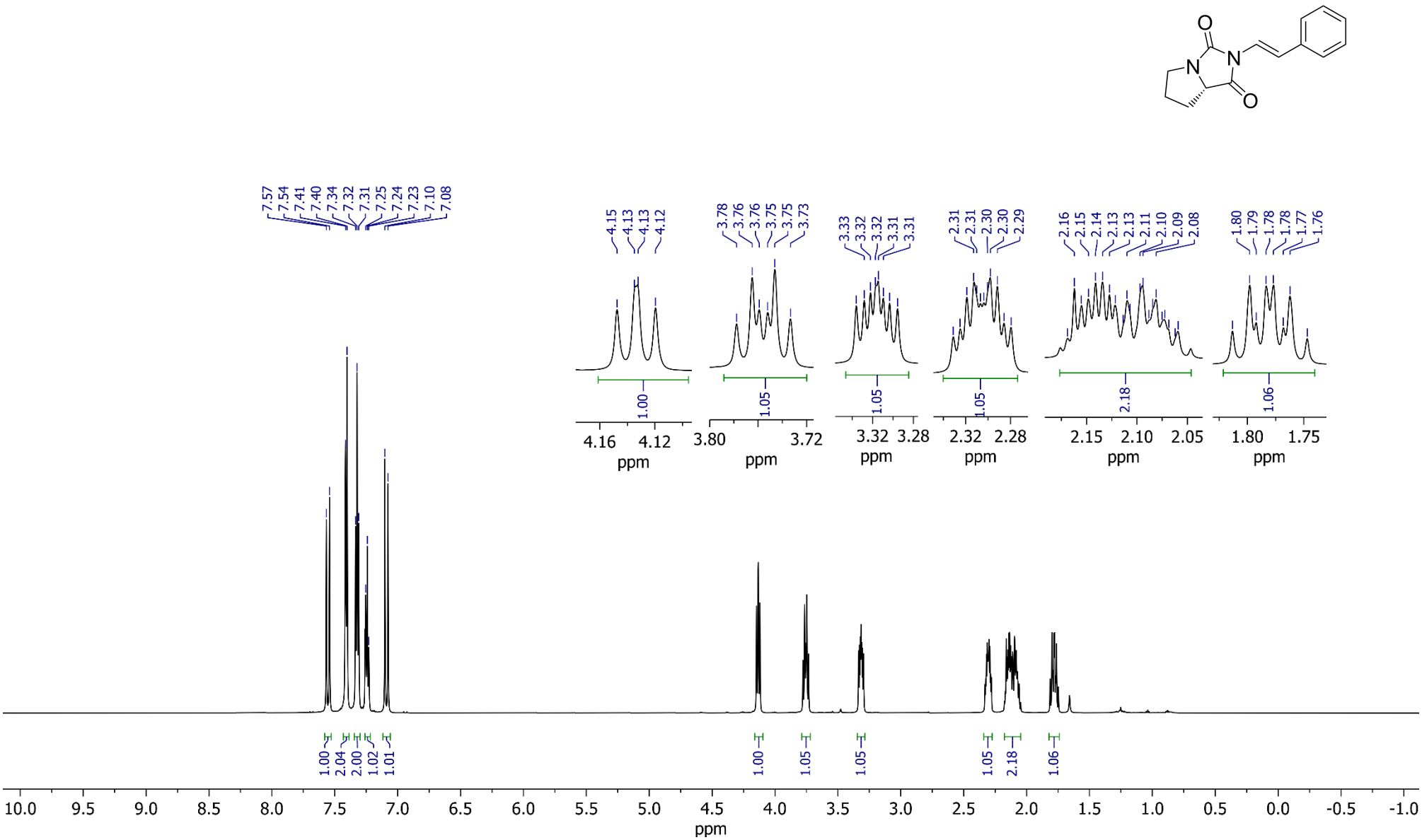
^{1}H NMR spectrum of 3a (600 MHz, $CDCl_3$)



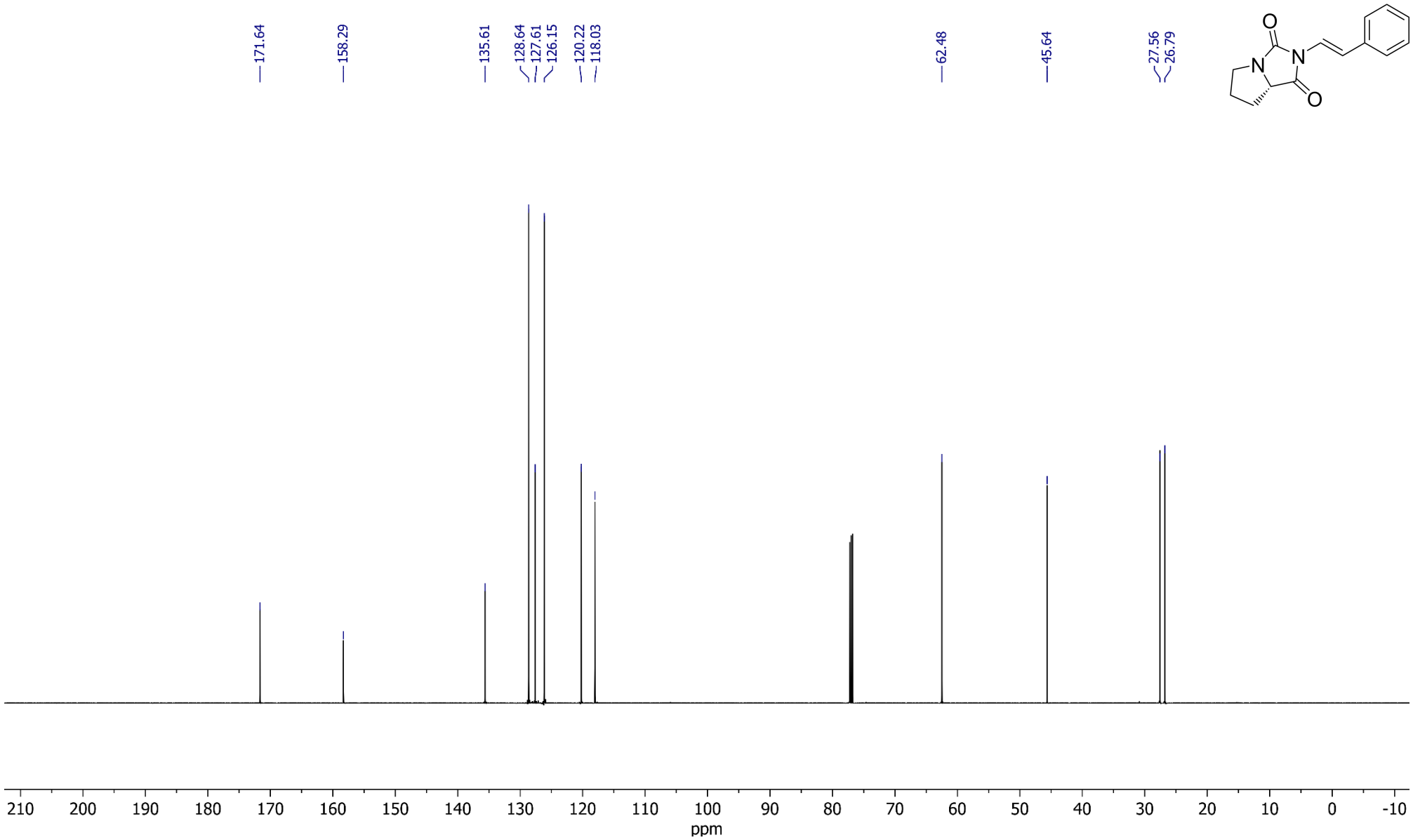
^{13}C NMR spectrum of 3a (151 MHz, $CDCl_3$)



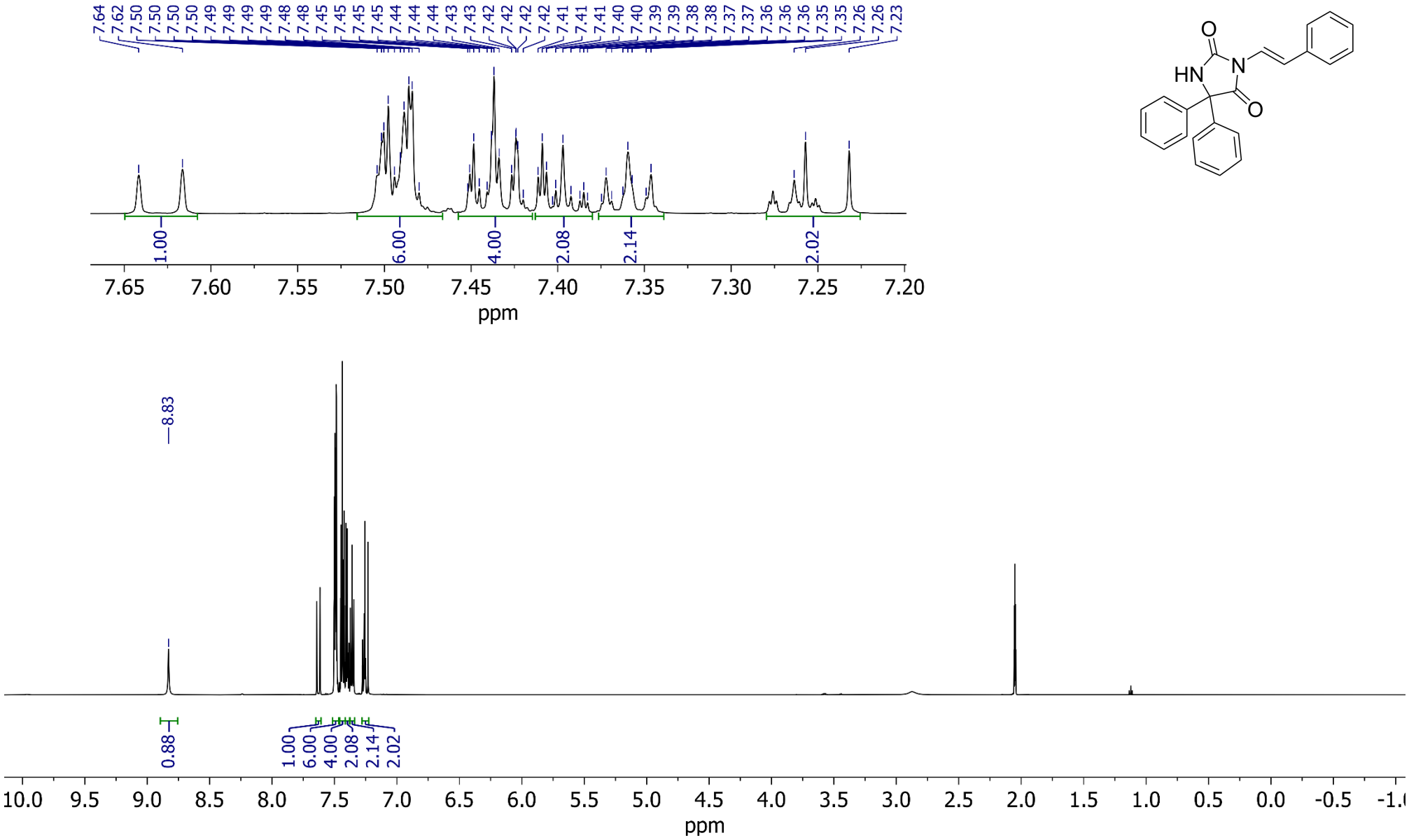
^{1}H NMR spectrum of 3b (600 MHz, $CDCl_3$)



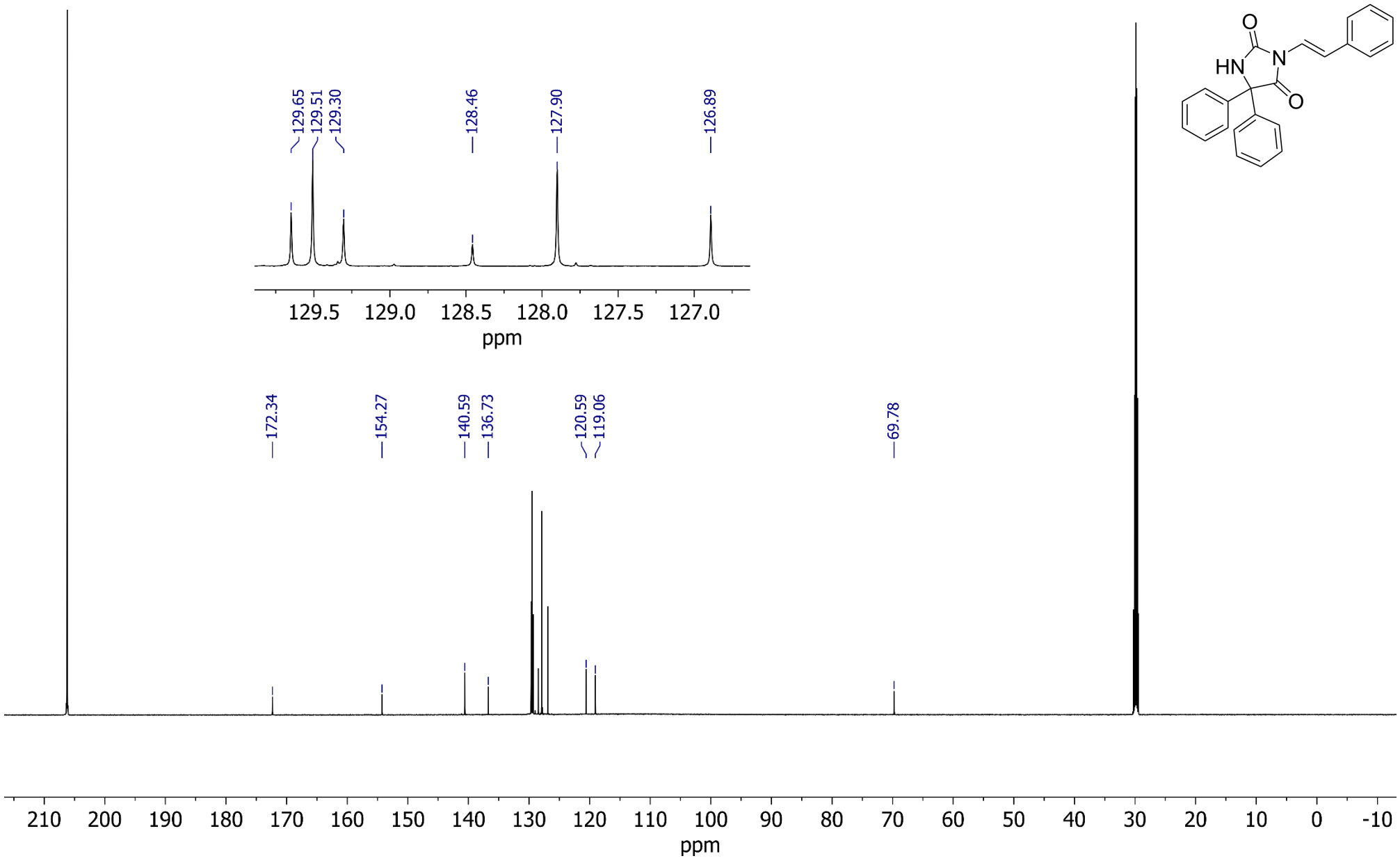
^{13}C NMR spectrum of 3b (151 MHz, $CDCl_3$)



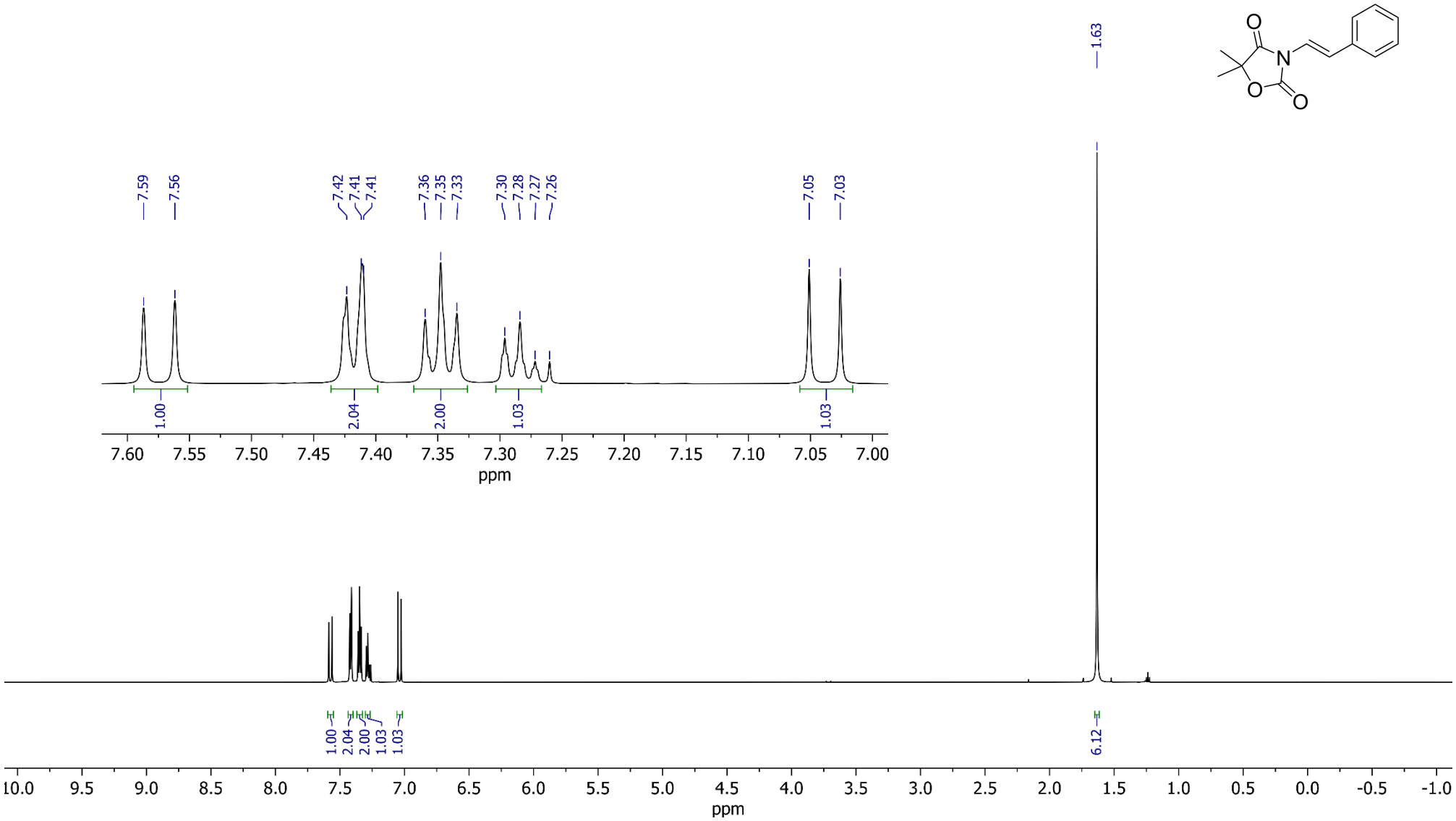
^{1}H NMR spectrum of 3c (600 MHz, Acetone-d_6)



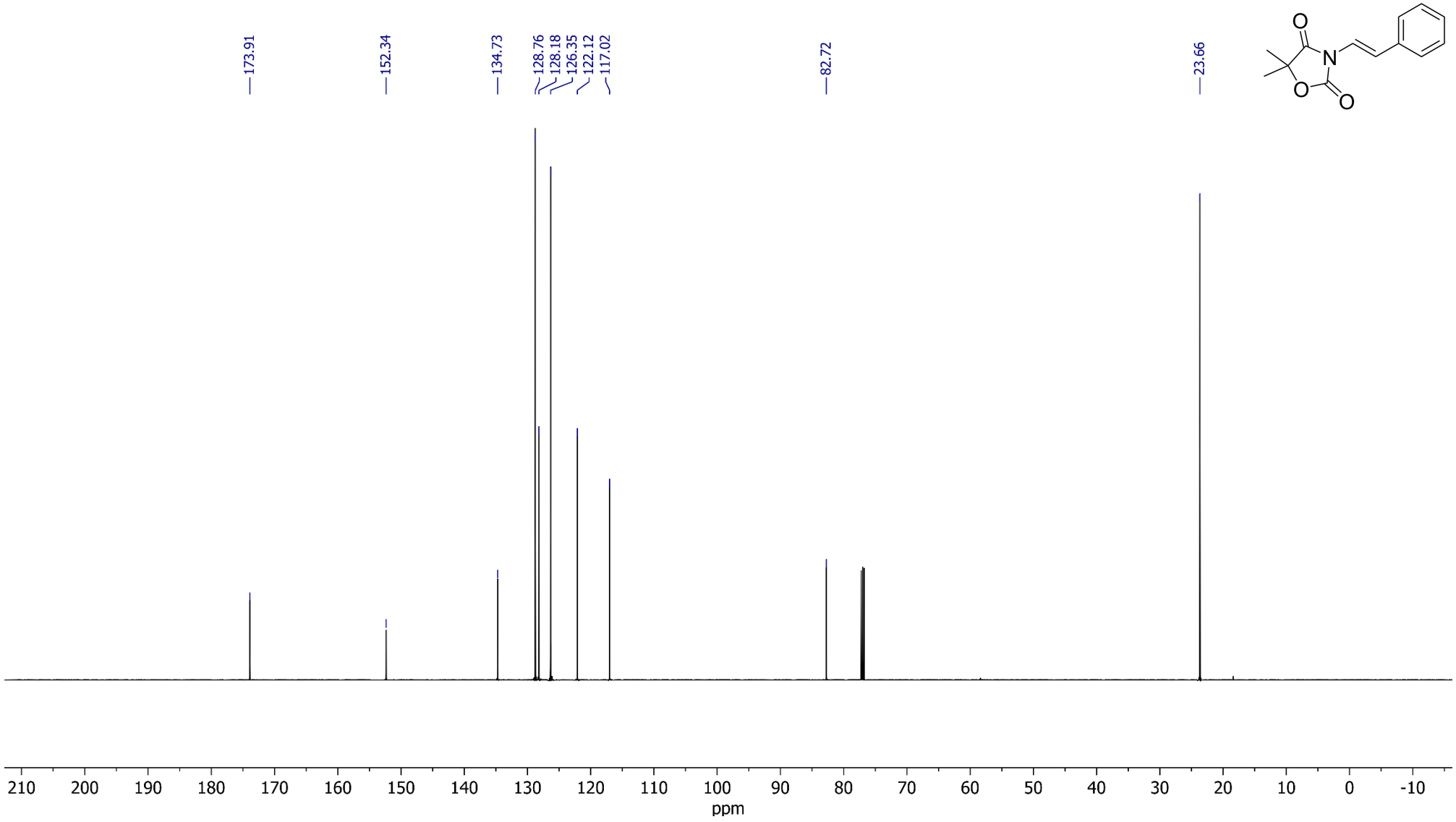
^{13}C NMR spectrum of 3c (151 MHz, Acetone-d_6)



^{1}H NMR spectrum of 3d (600 MHz, $CDCl_3$)



^{13}C NMR spectrum of 3d (151 MHz, $CDCl_3$)



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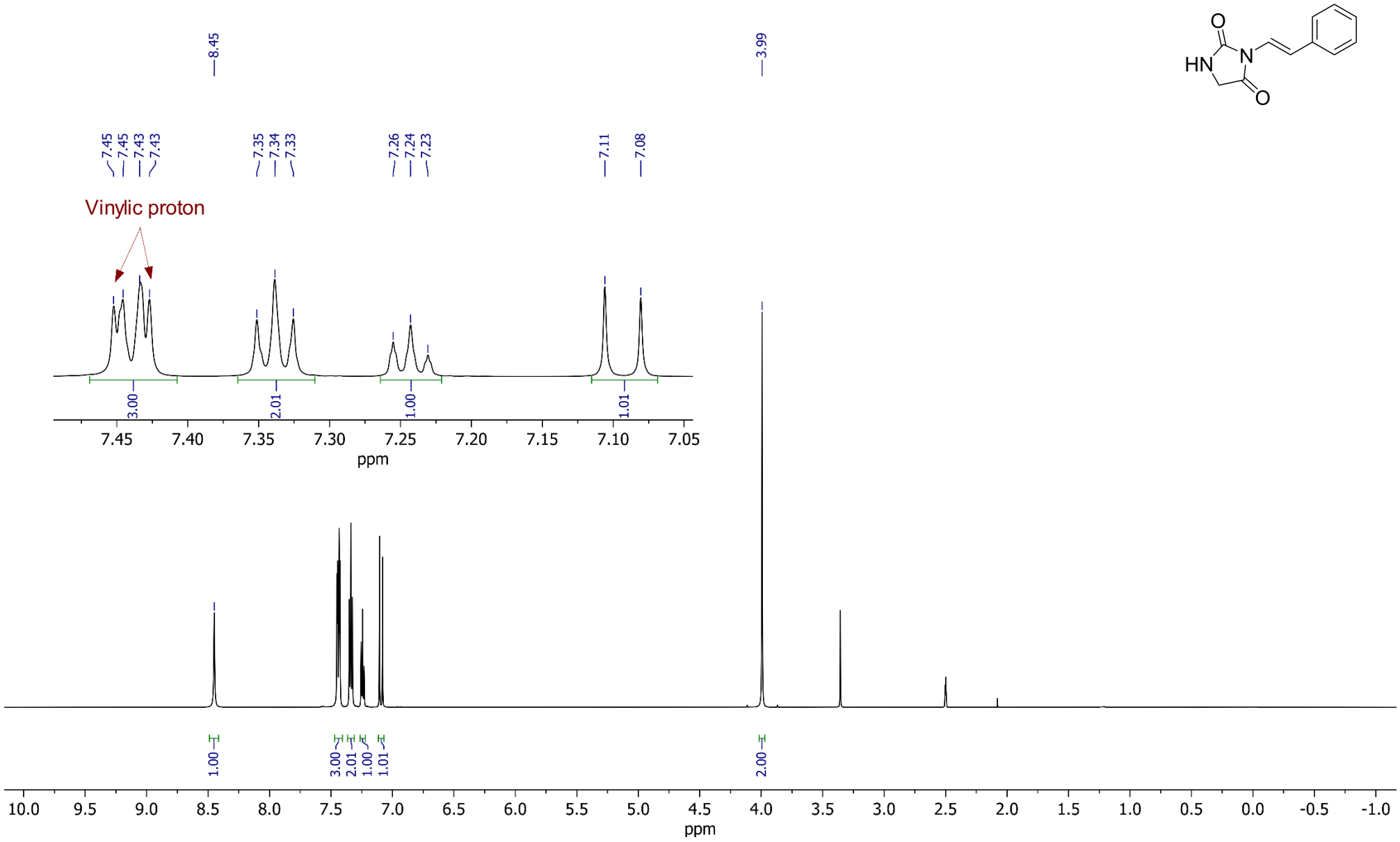
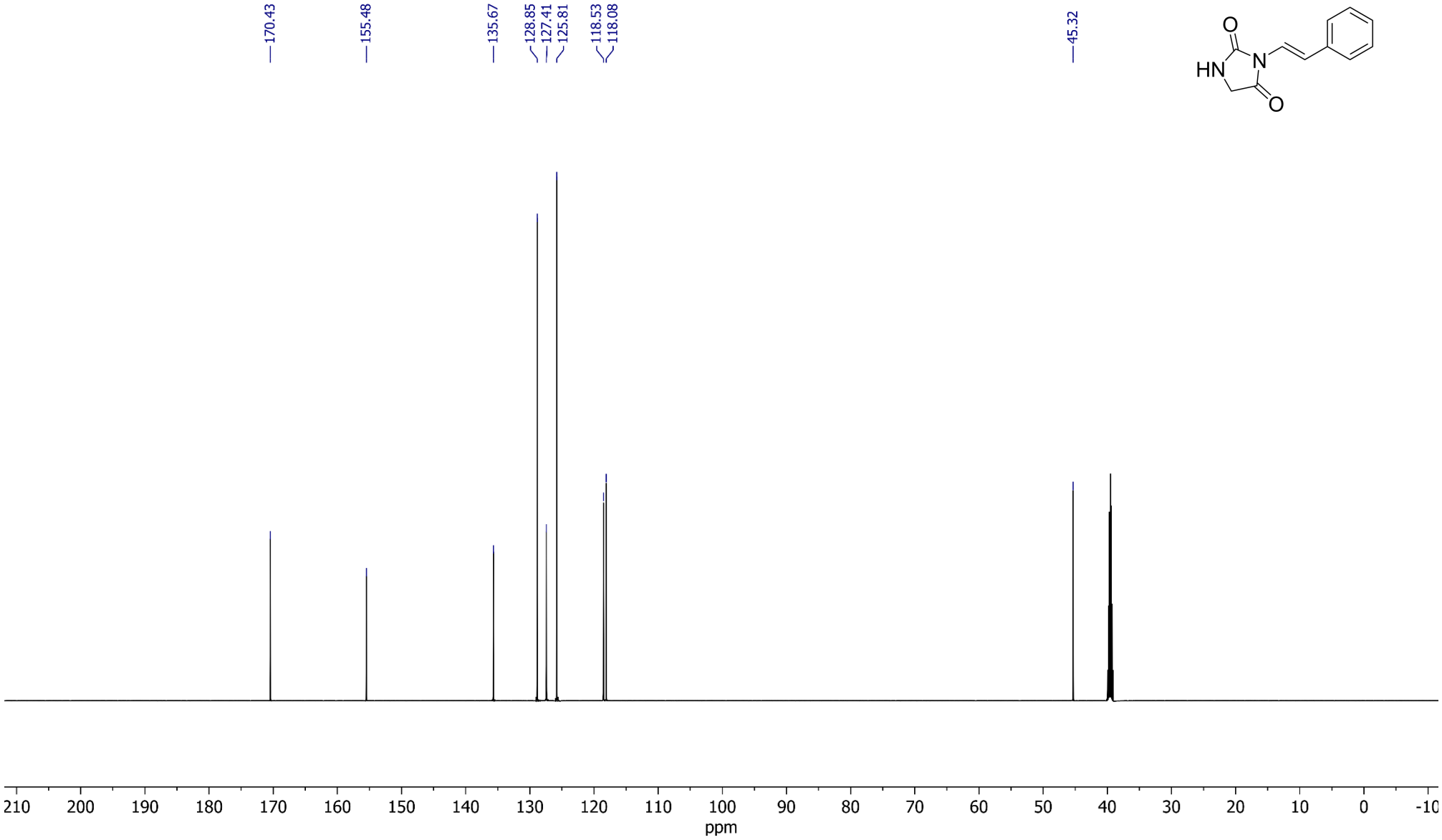
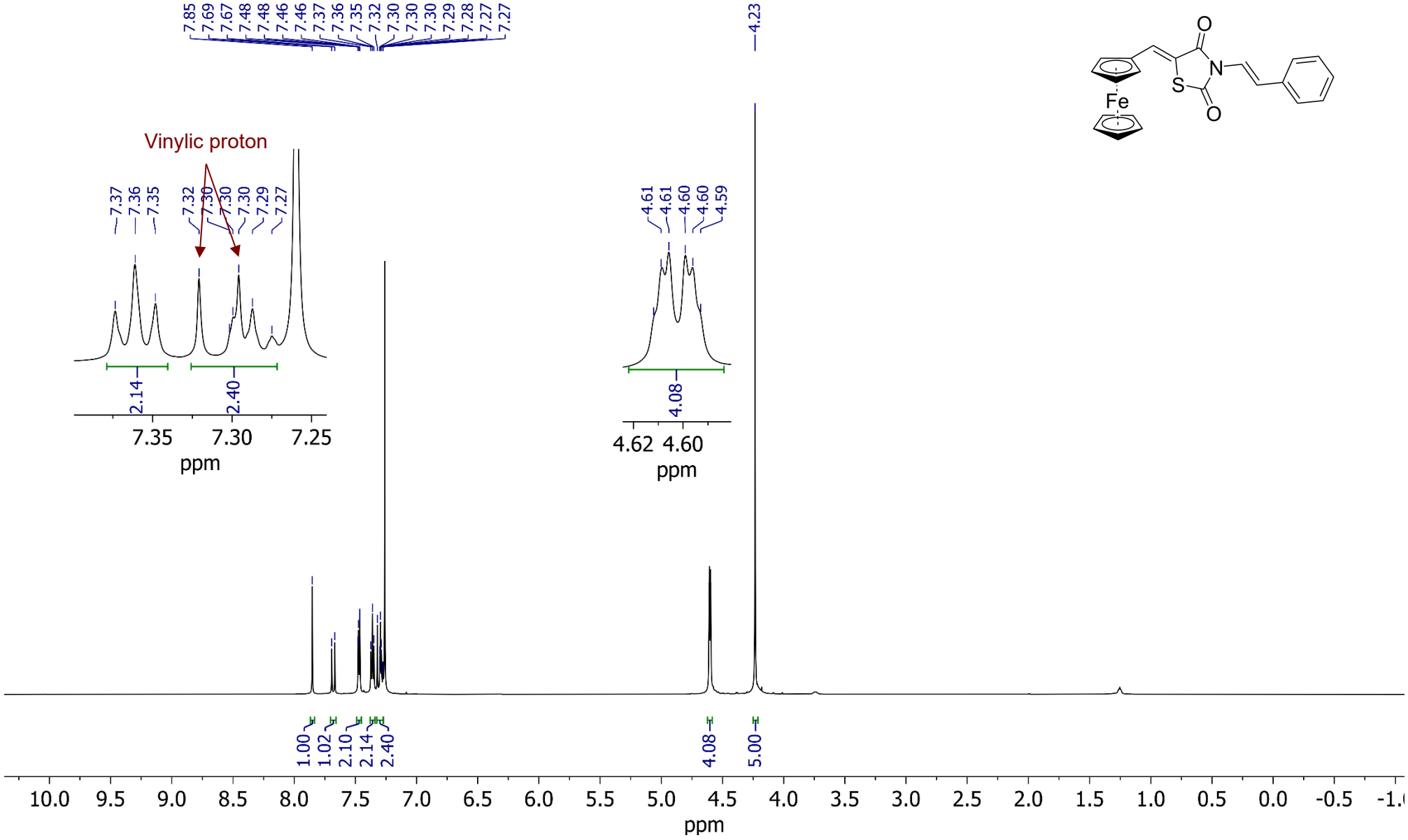


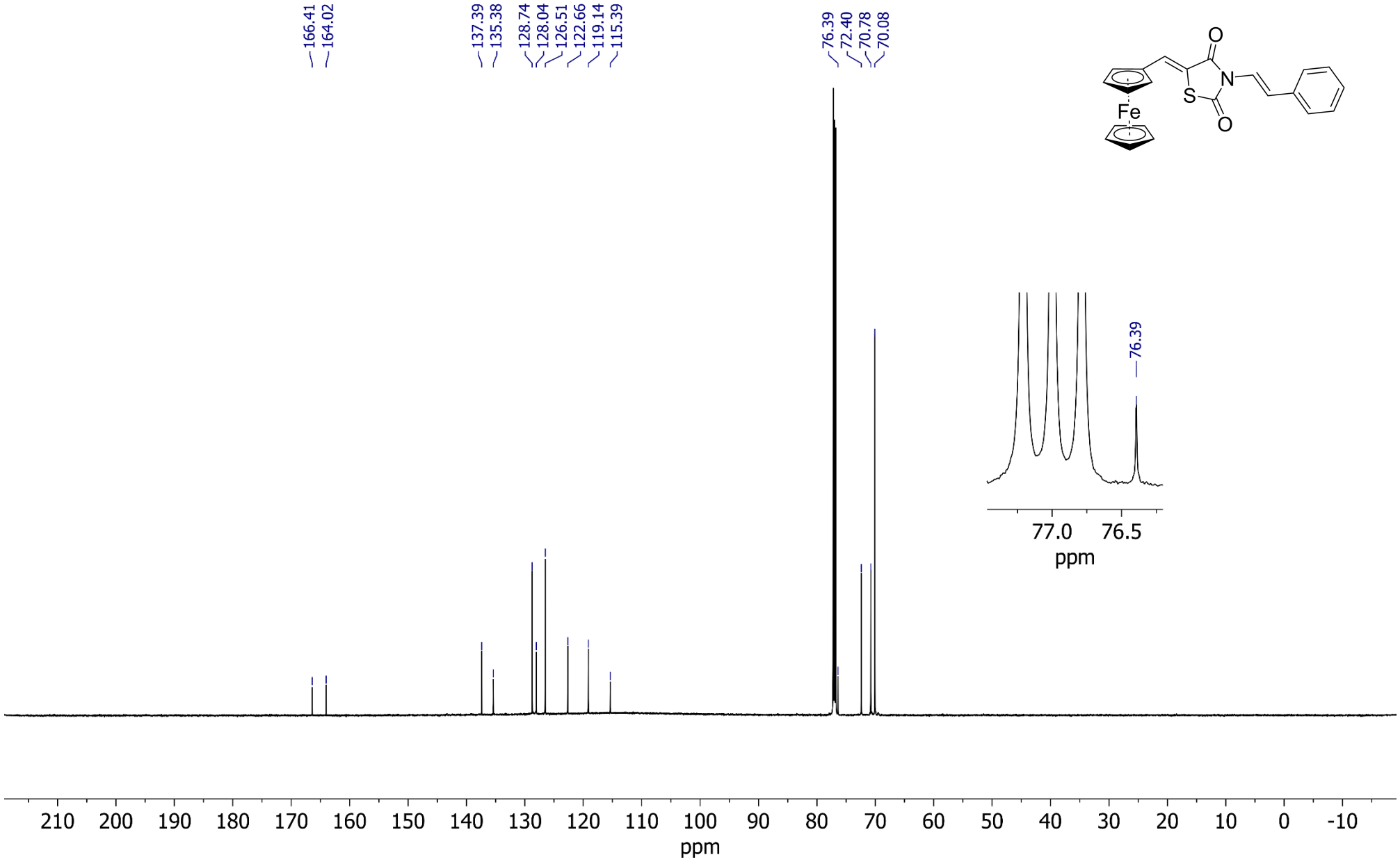
^{13}C NMR spectrum of 3e (151 MHz, DMSO-d_6)



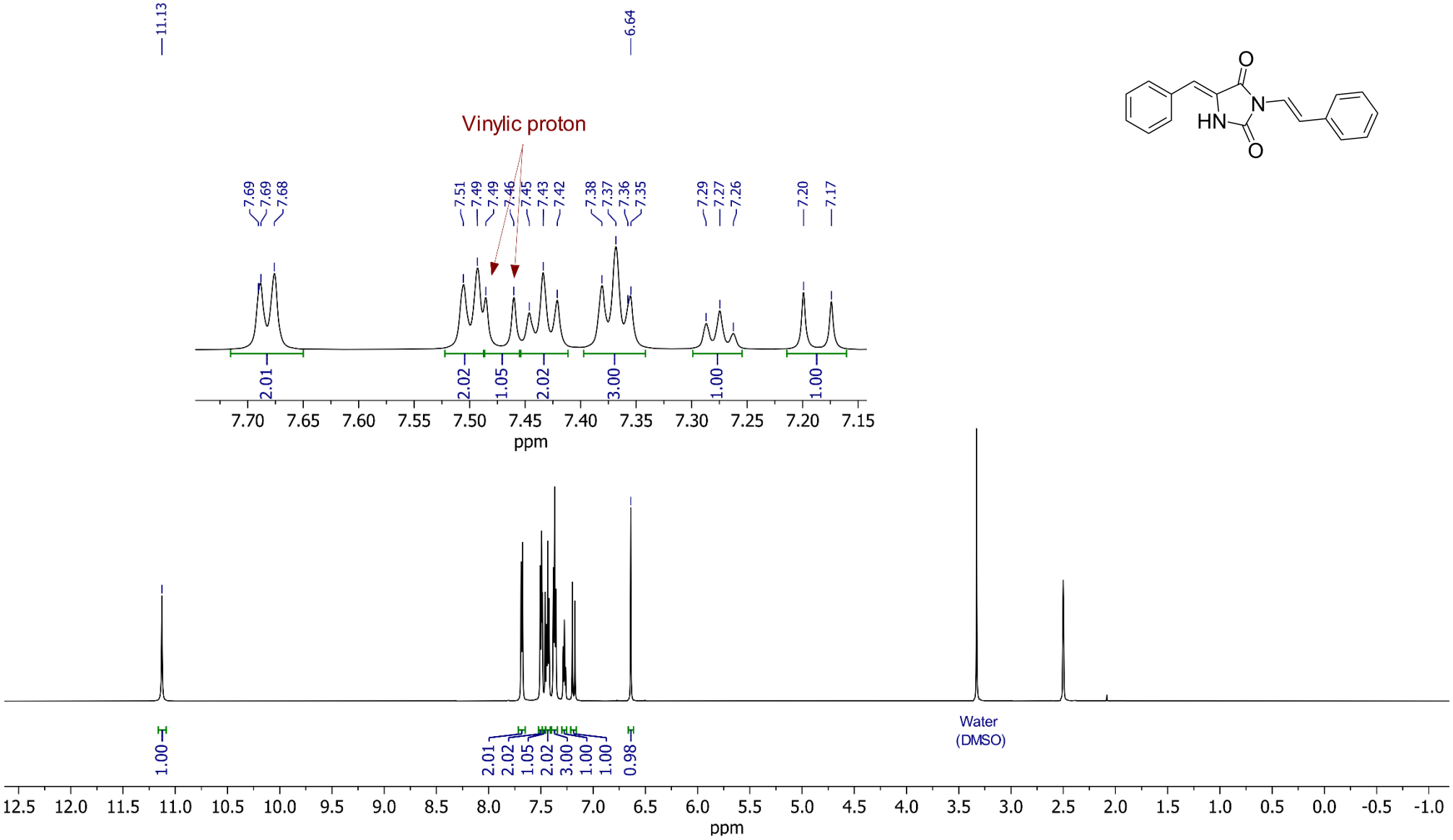
^{1}H NMR spectrum of 3f (600 MHz, $CDCl_3$)



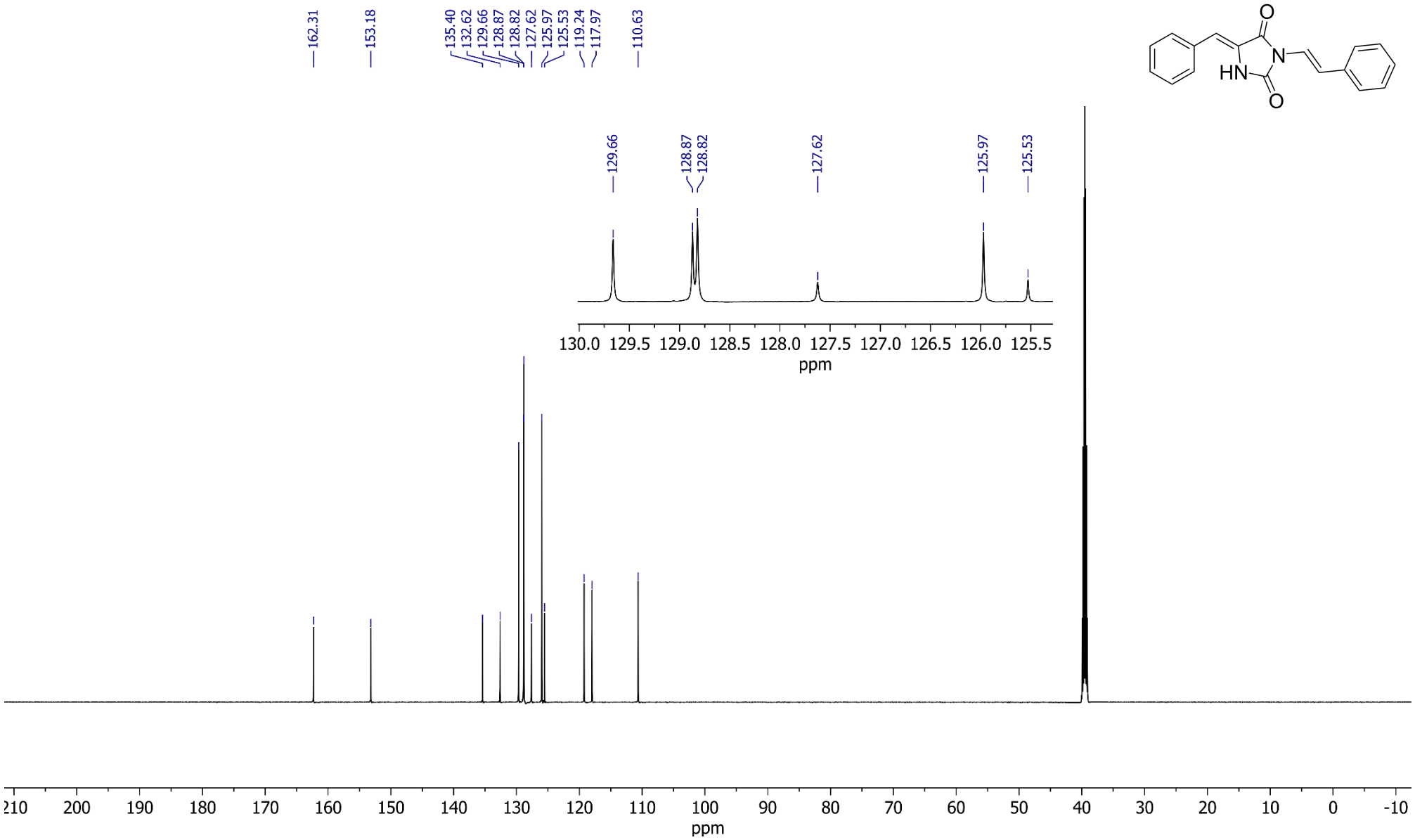
^{13}C NMR spectrum of 3f (151 MHz, $CDCl_3$)



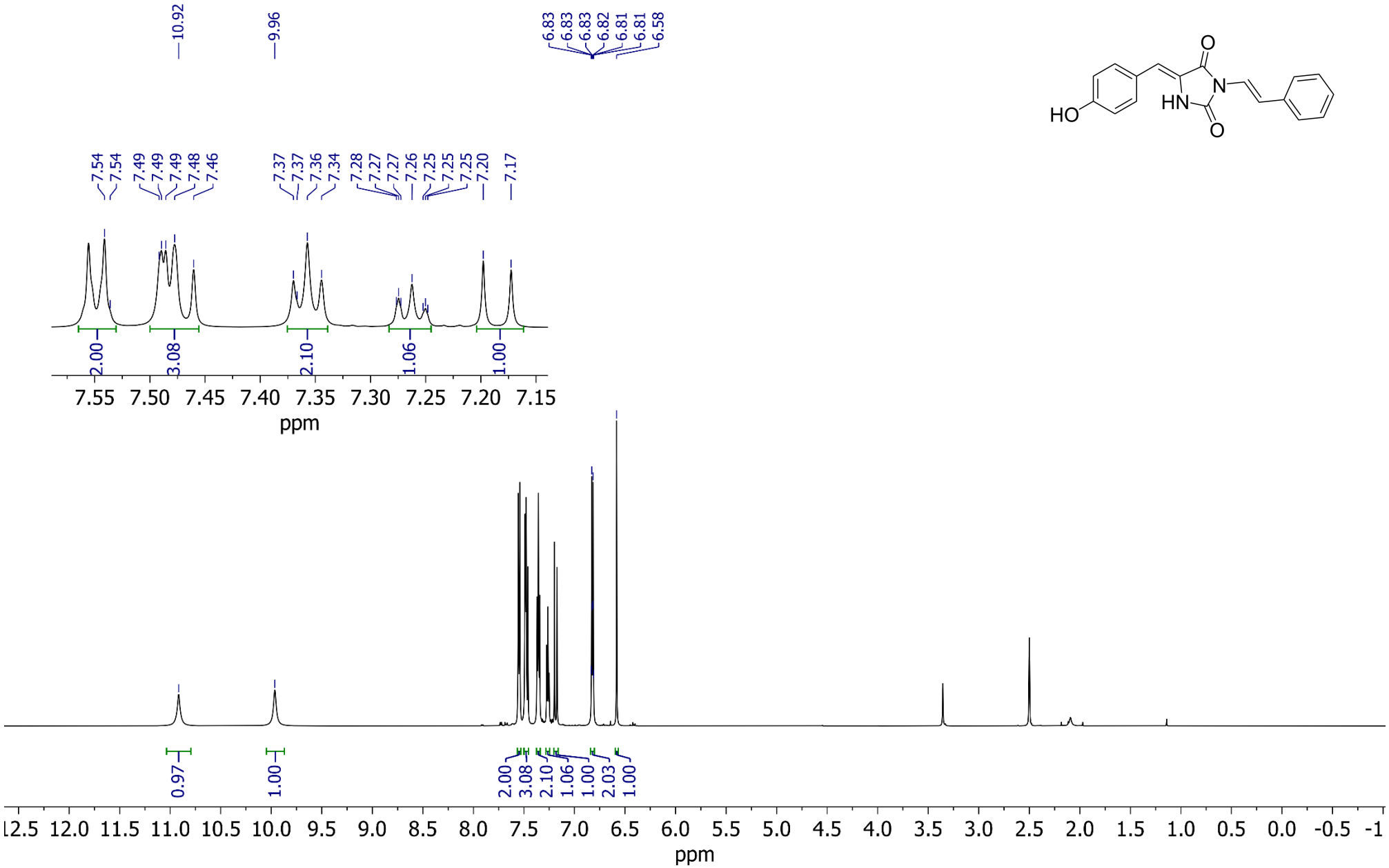
^{1}H NMR spectrum of 3g (600 MHz, DMSO-d_6)



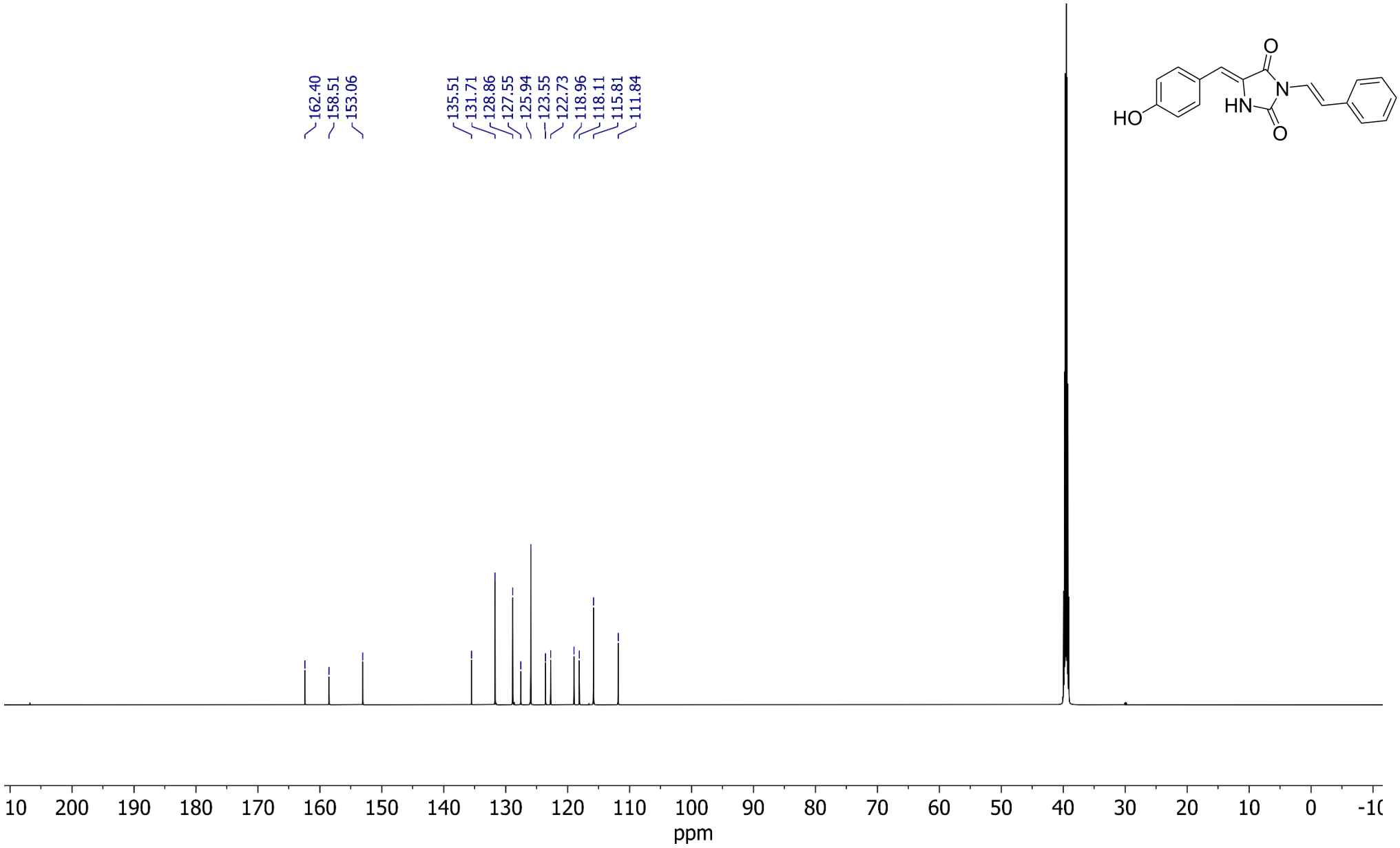
^{13}C NMR spectrum of 3g (151 MHz, DMSO-d_6)



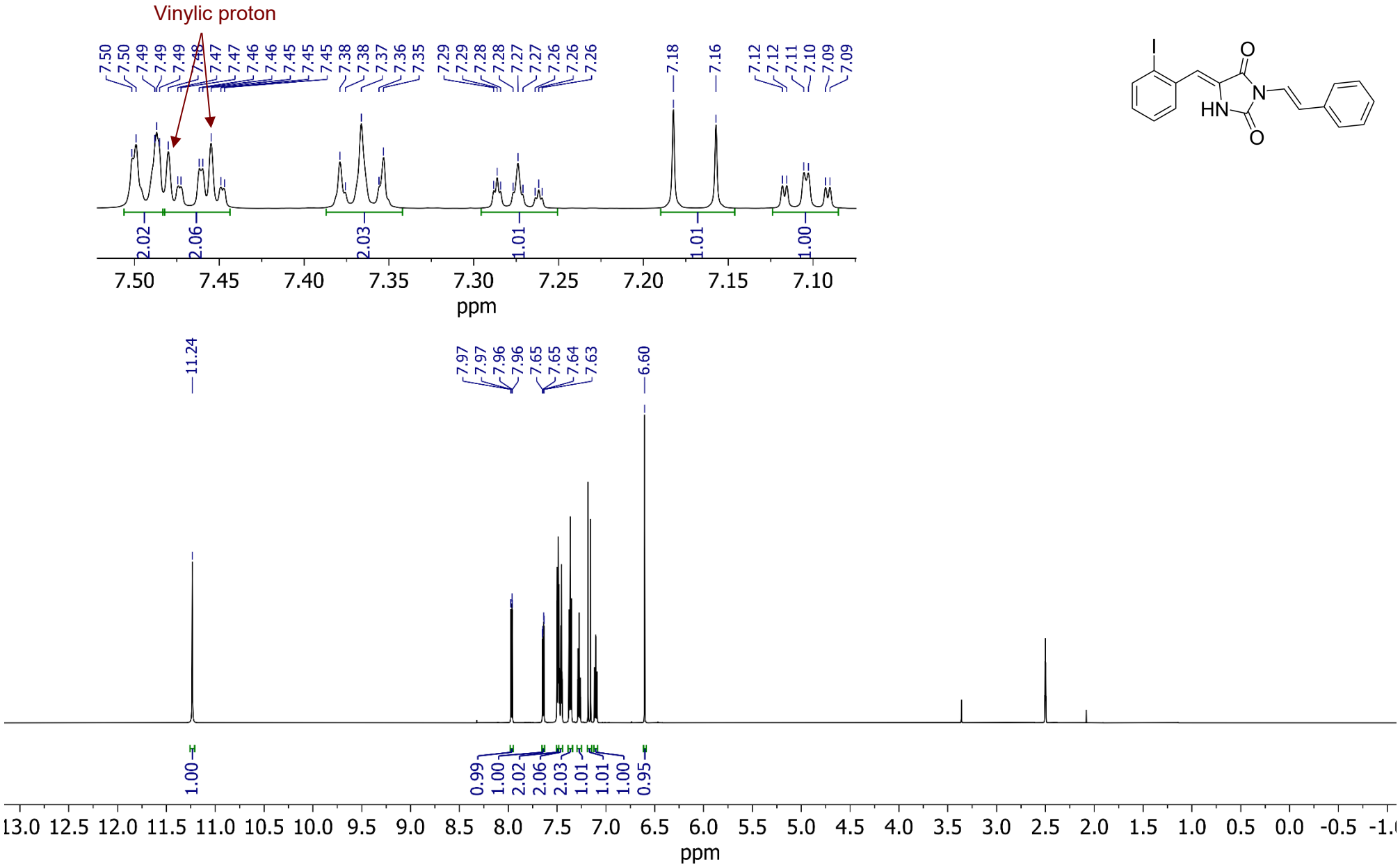
^{1}H NMR spectrum of 3h (600 MHz, DMSO-d_6)



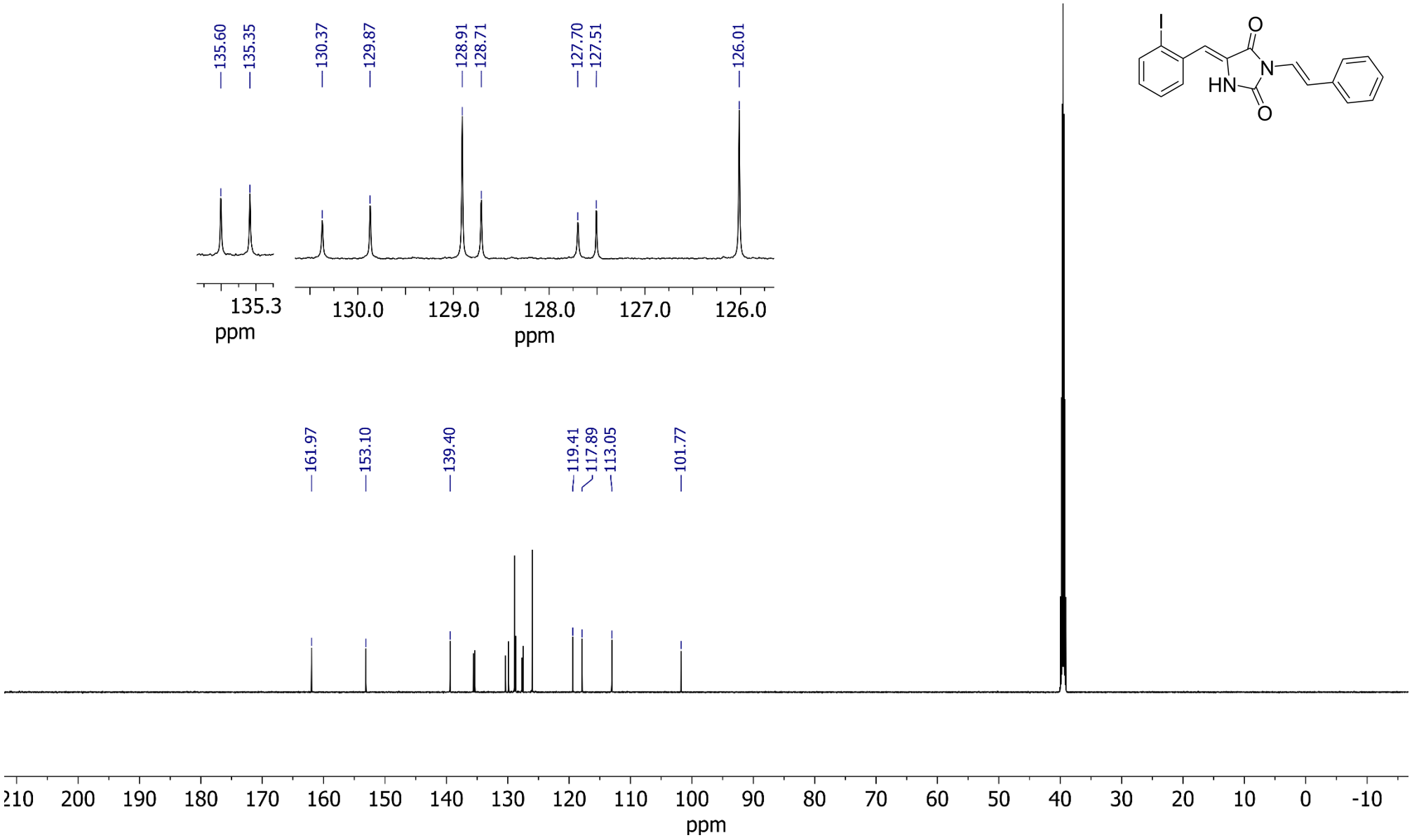
^{13}C NMR spectrum of 3h (600 MHz, DMSO-d_6)



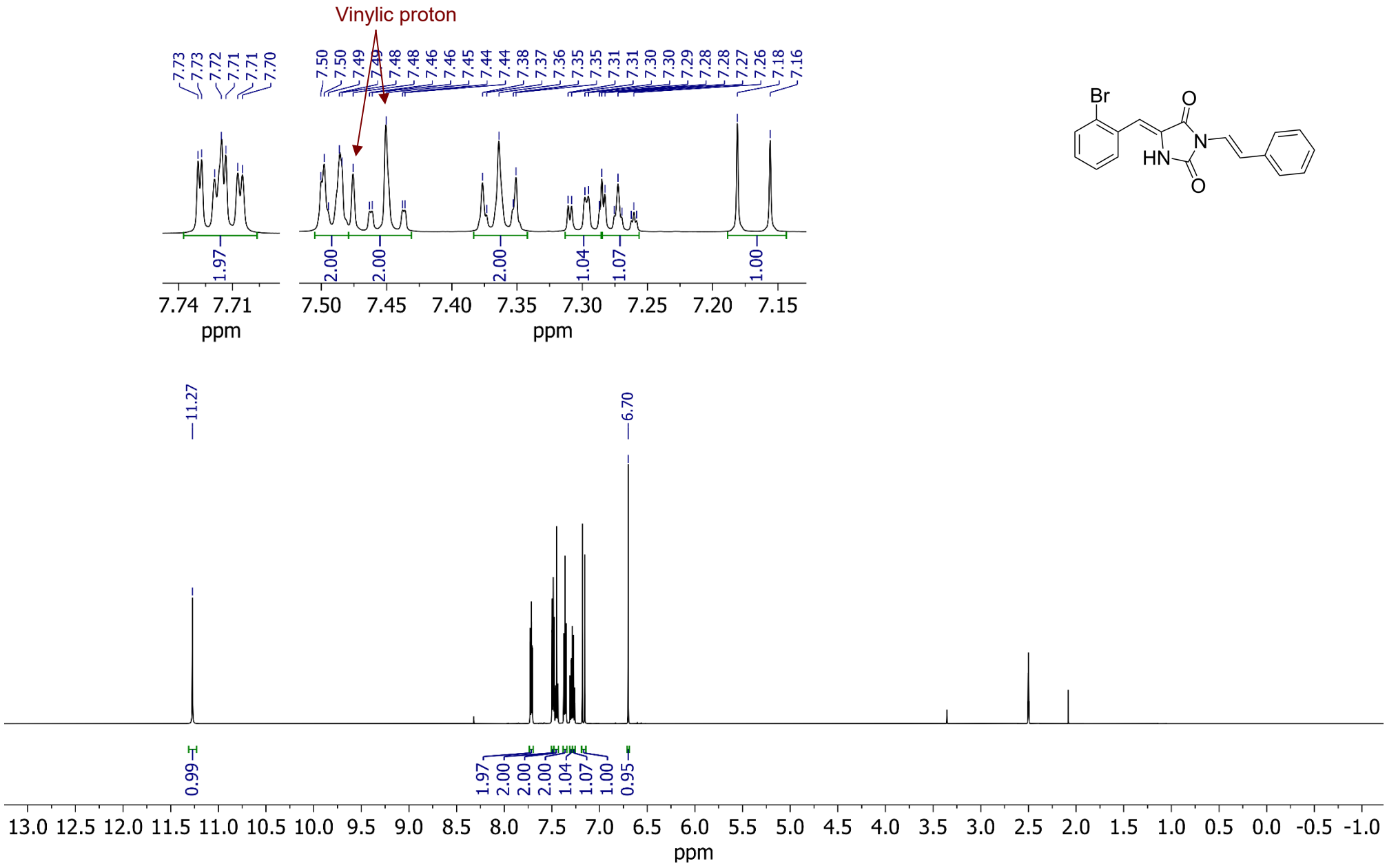
^{1}H NMR spectrum of 3i (600 MHz, DMSO-d_6)



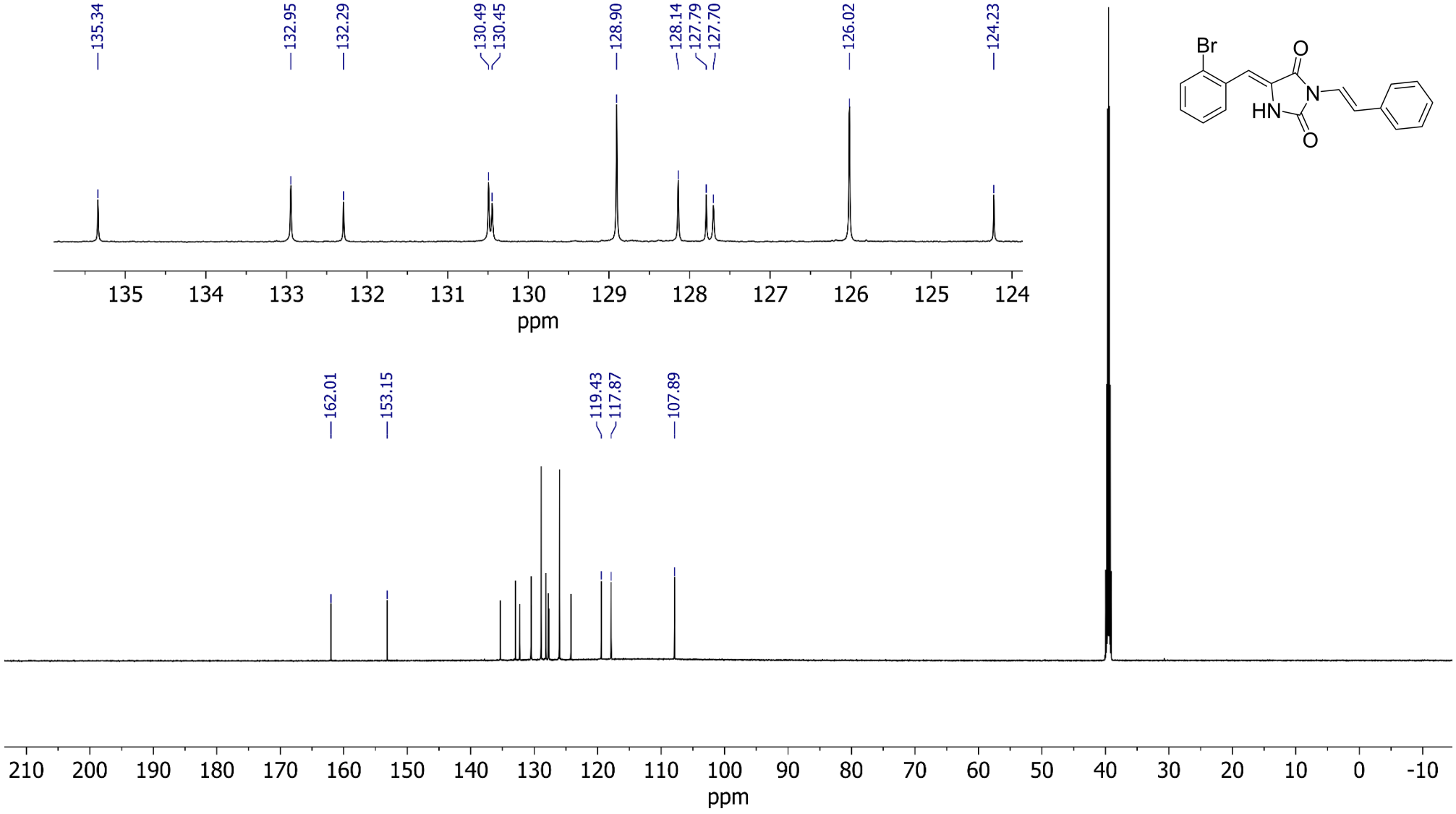
^{13}C NMR spectrum of 3i (600 MHz, DMSO-d_6)



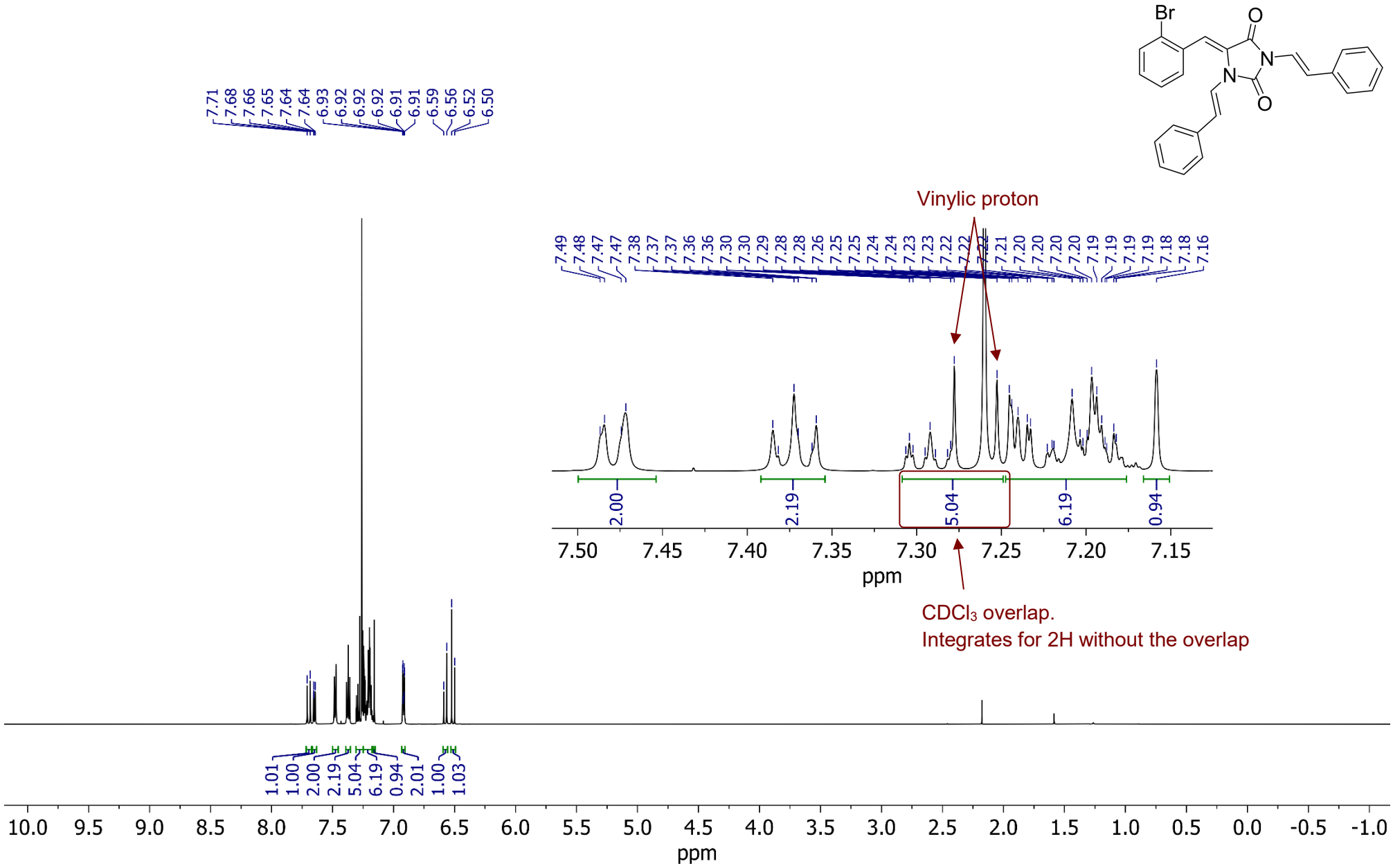
^{1}H NMR spectrum of 3j (600 MHz, DMSO-d_6)



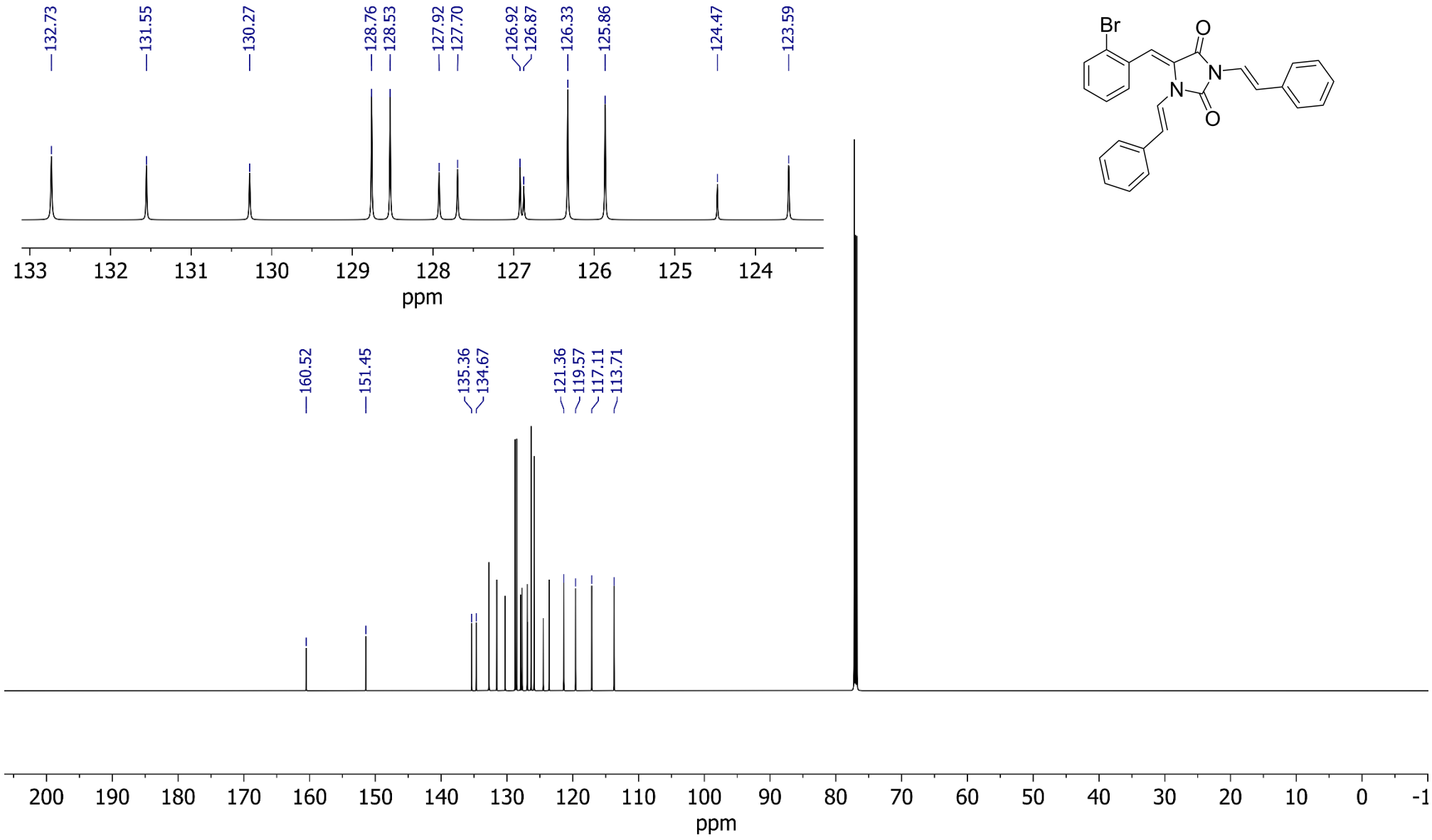
^{13}C NMR spectrum of 3j (600 MHz, DMSO-d_6)



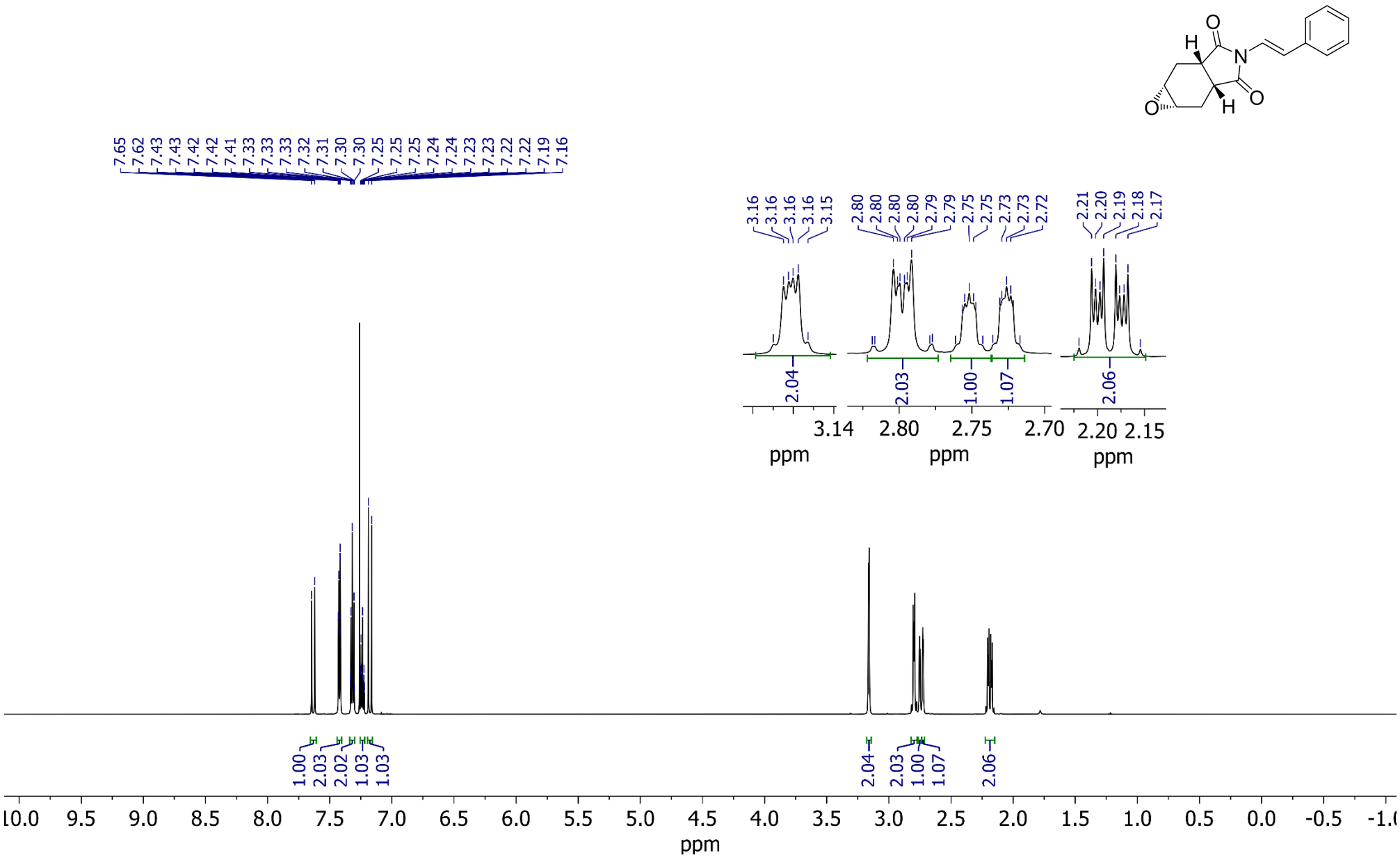
^{1}H NMR spectrum of 3j' (600 MHz, $CDCl_3$)



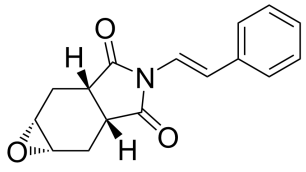
^{13}C NMR spectrum of 3j' (151 MHz, $CDCl_3$)

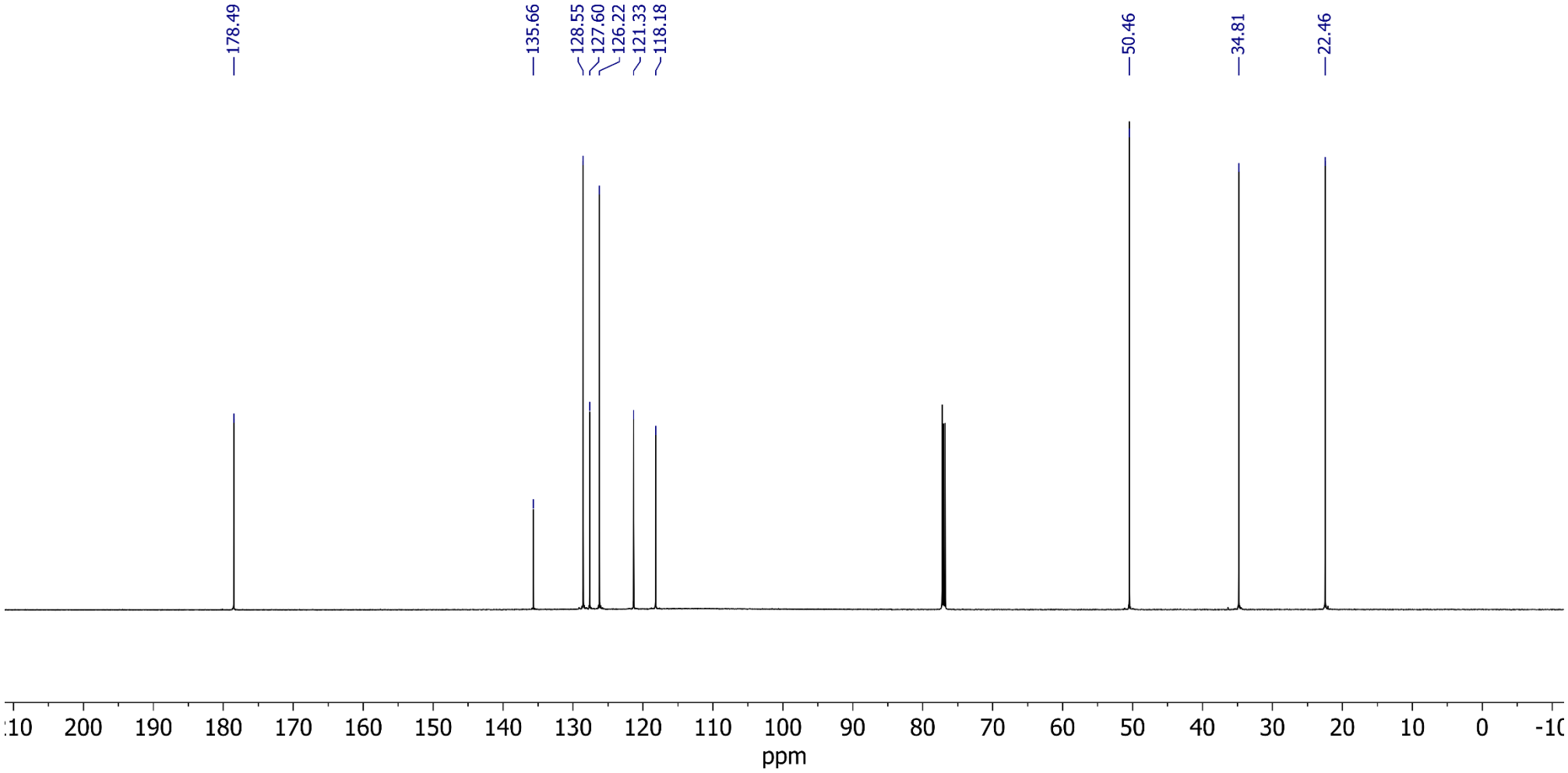


^{1}H NMR spectrum of 3k (600 MHz, $CDCl_3$)

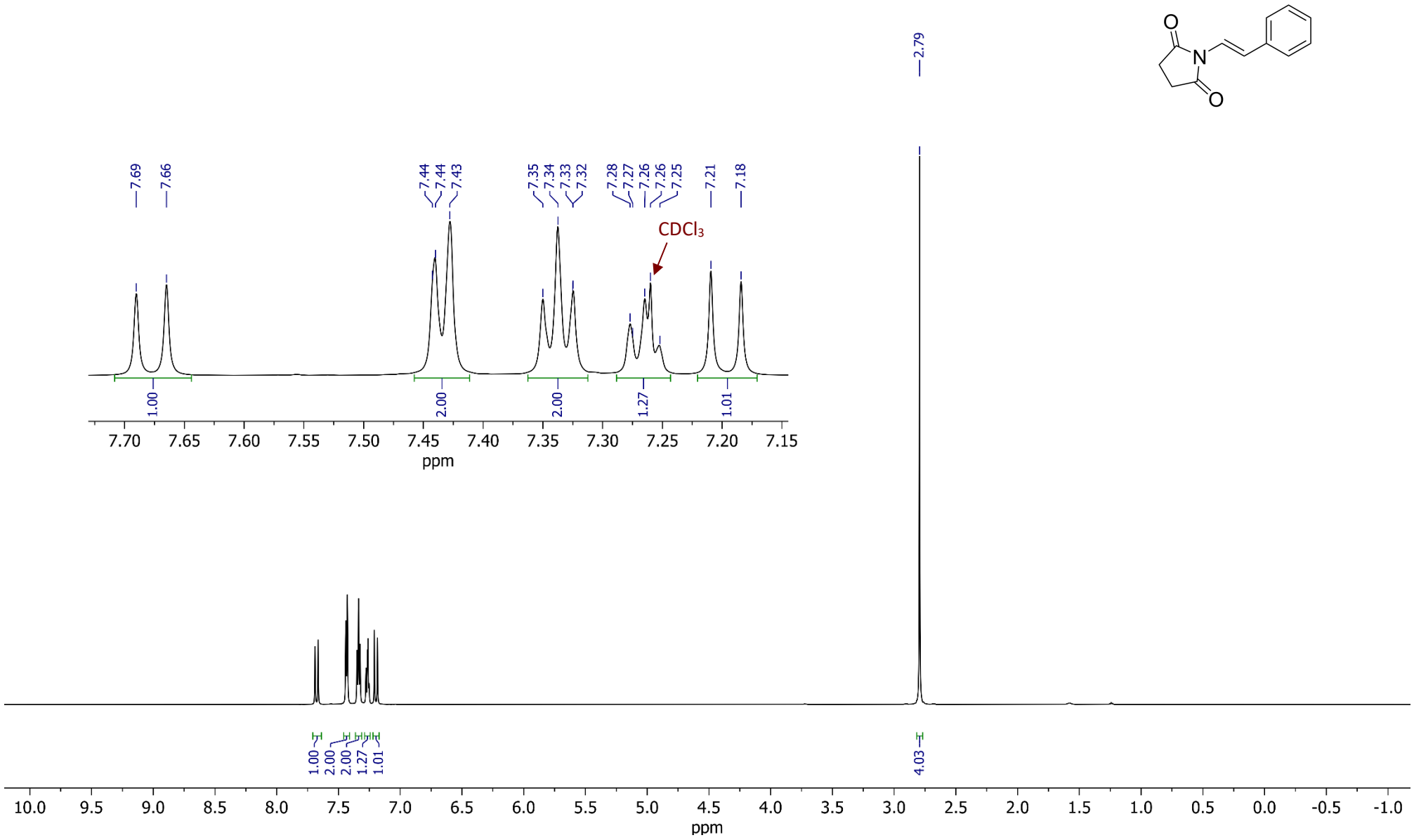


^{13}C NMR spectrum of 3k (151 MHz, $CDCl_3$)

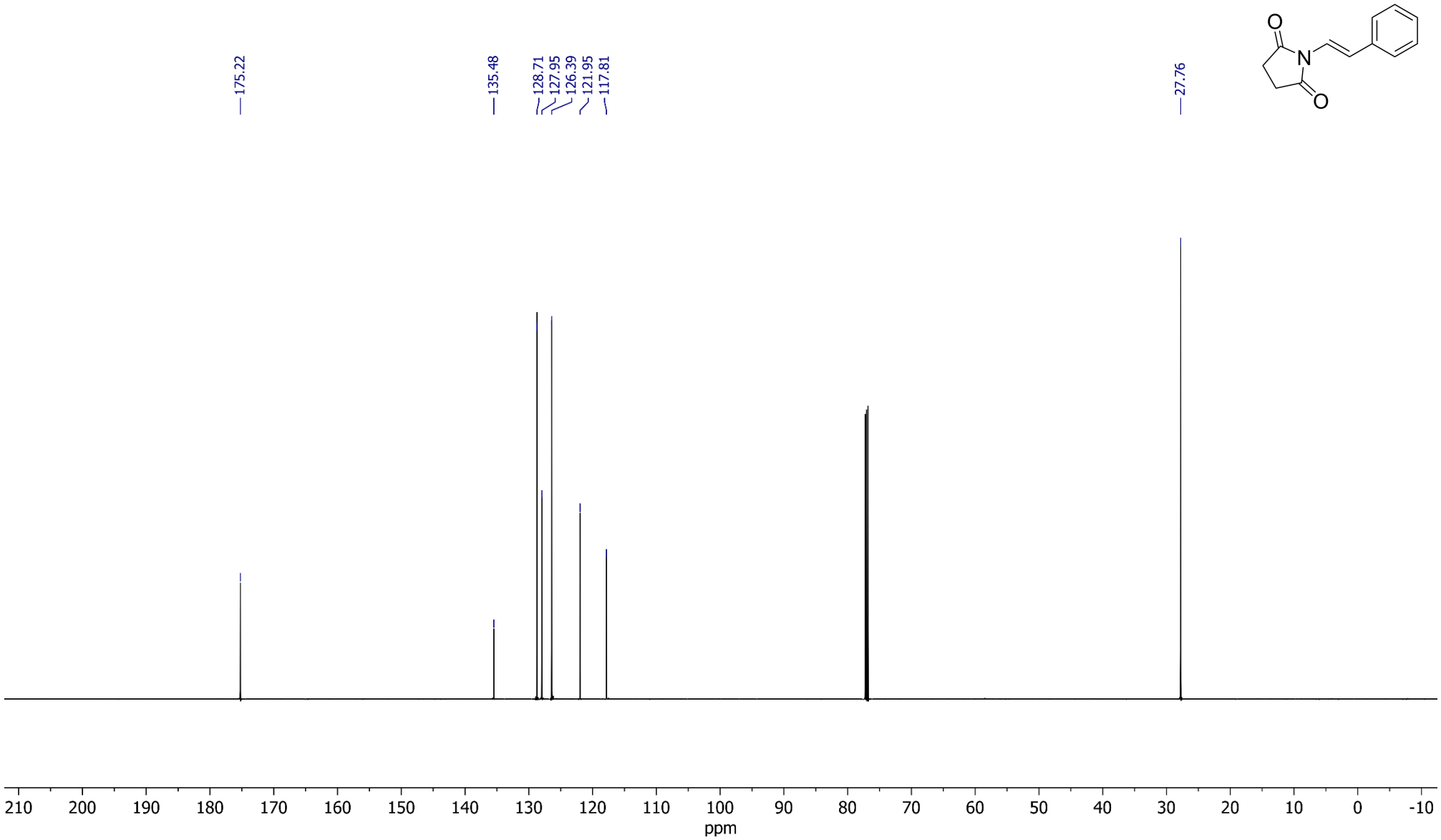




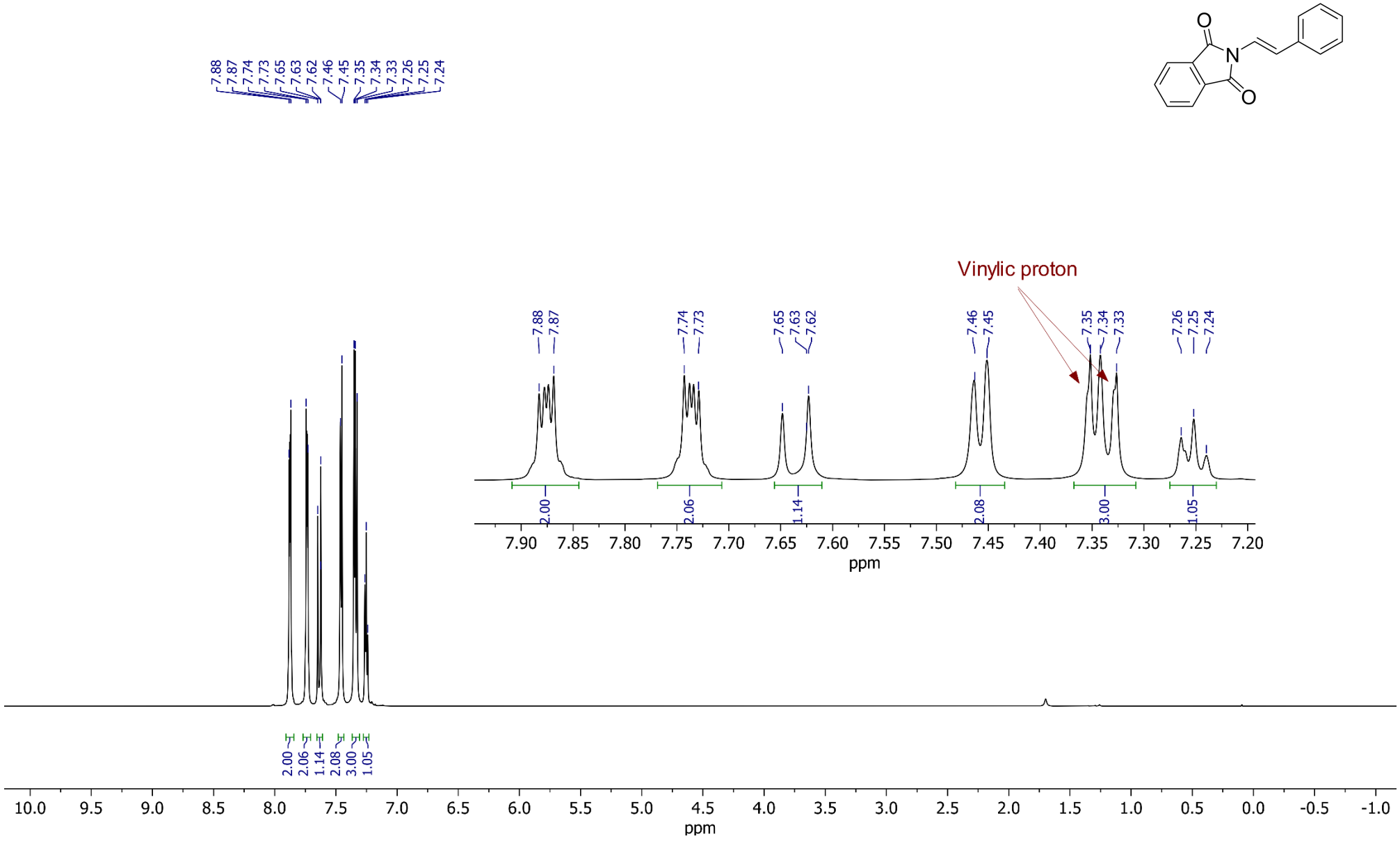
^{1}H NMR spectrum of 3l (600 MHz, $CDCl_3$)



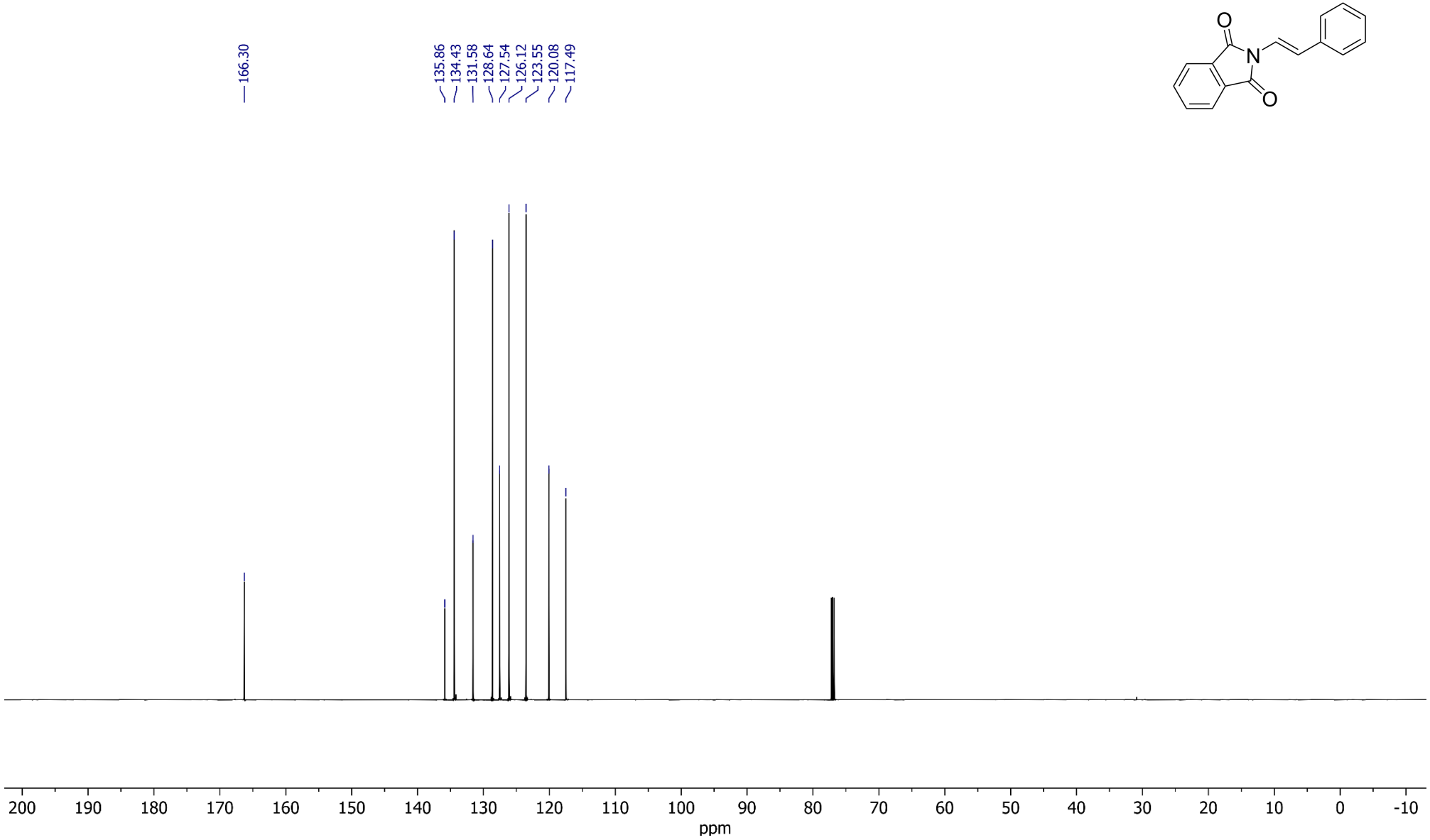
^{13}C NMR spectrum of 3l (151 MHz, $CDCl_3$)



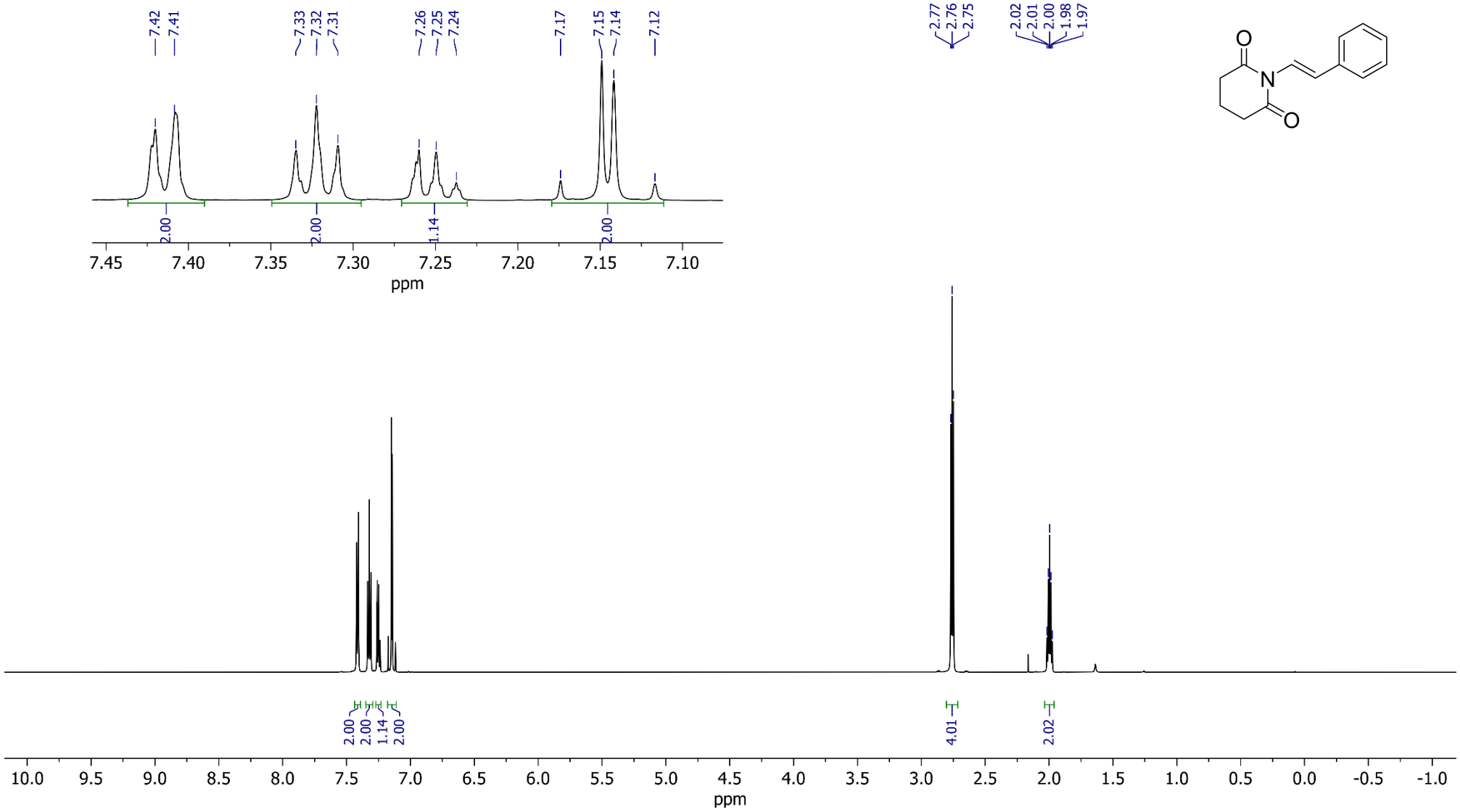
^{1}H NMR spectrum of 3m (600 MHz, $CDCl_3$)



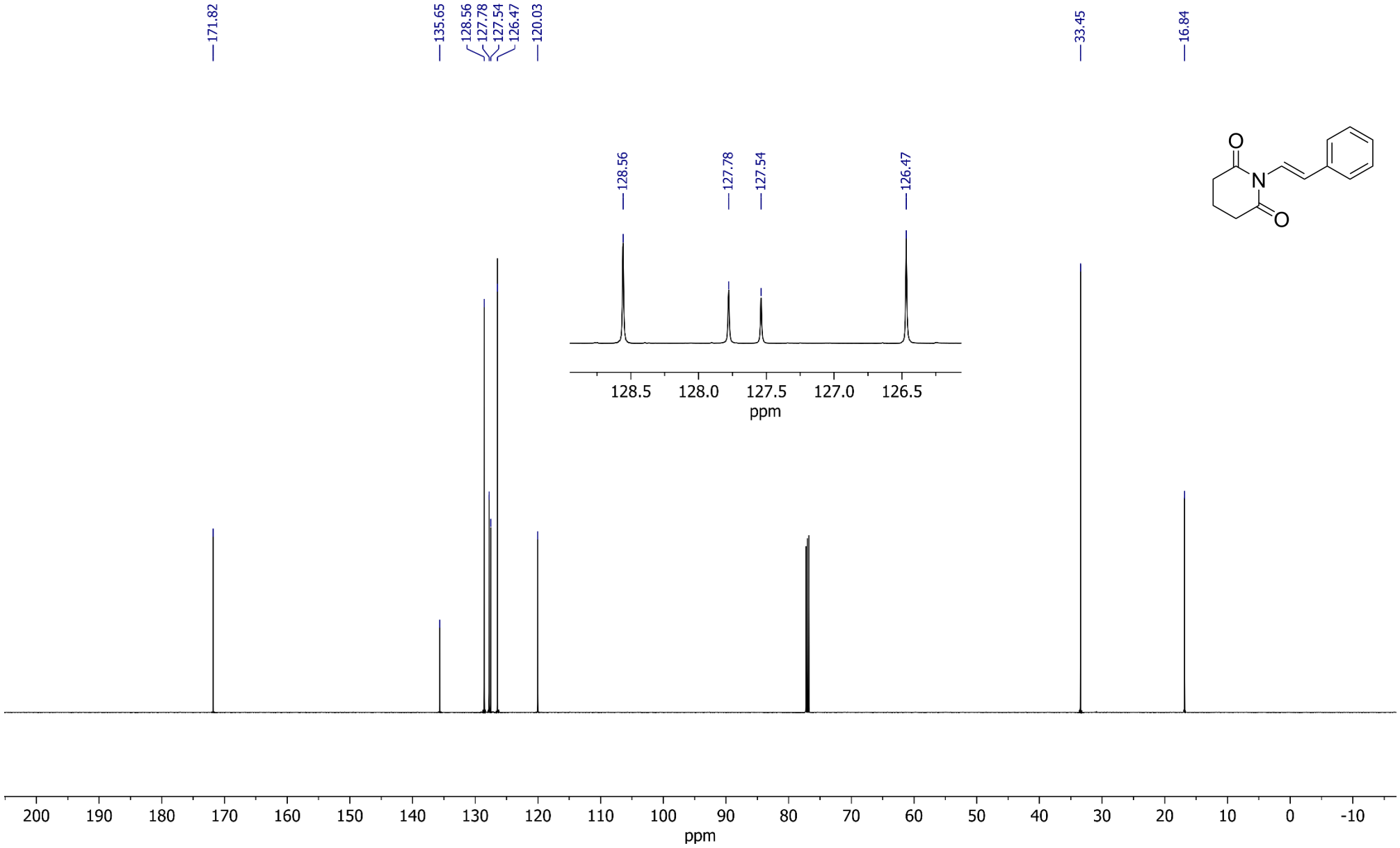
^{13}C NMR spectrum of 3m (151 MHz, $CDCl_3$)



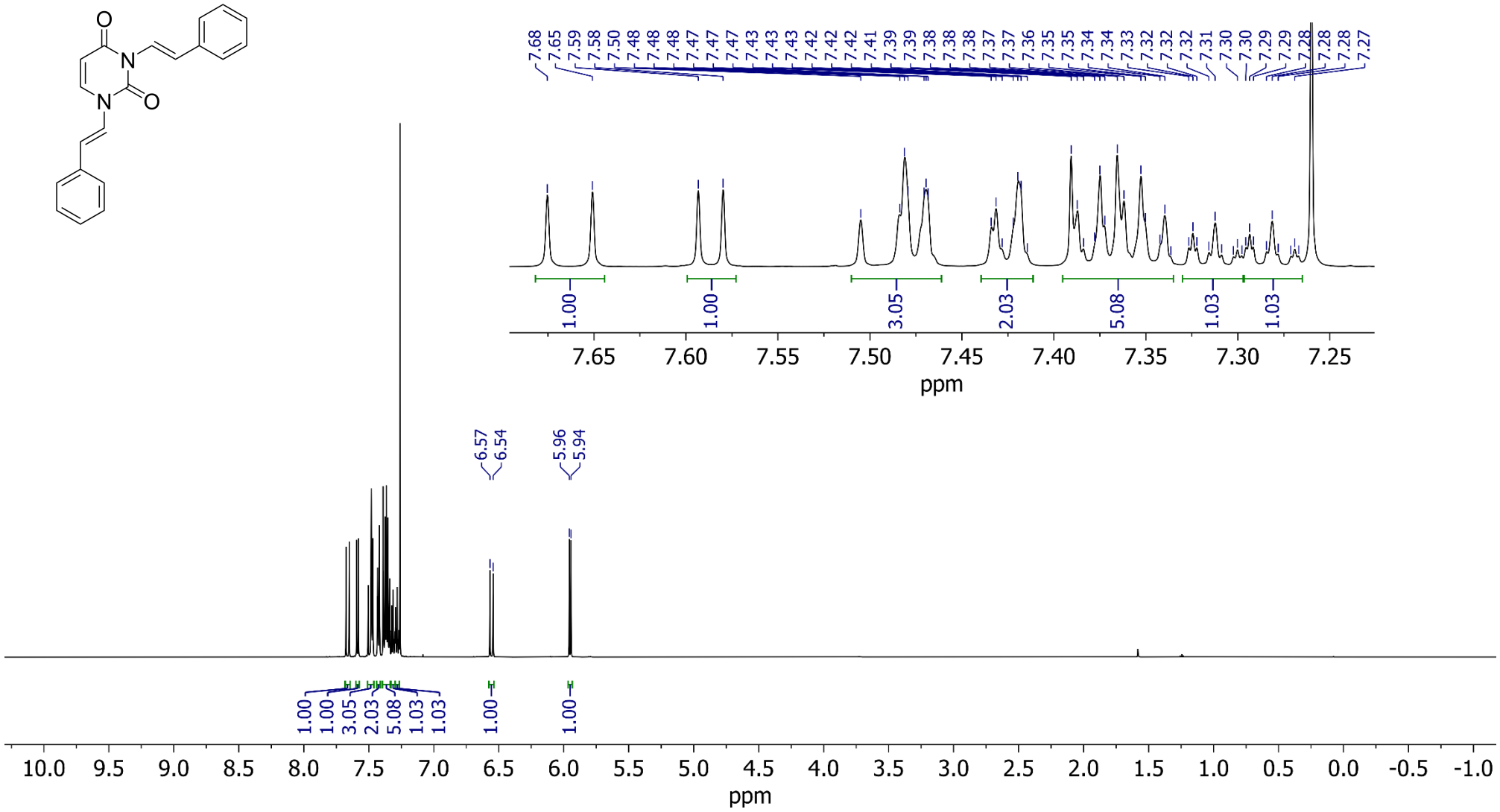
^{1}H NMR spectrum of 3p (600 MHz, $CDCl_3$)



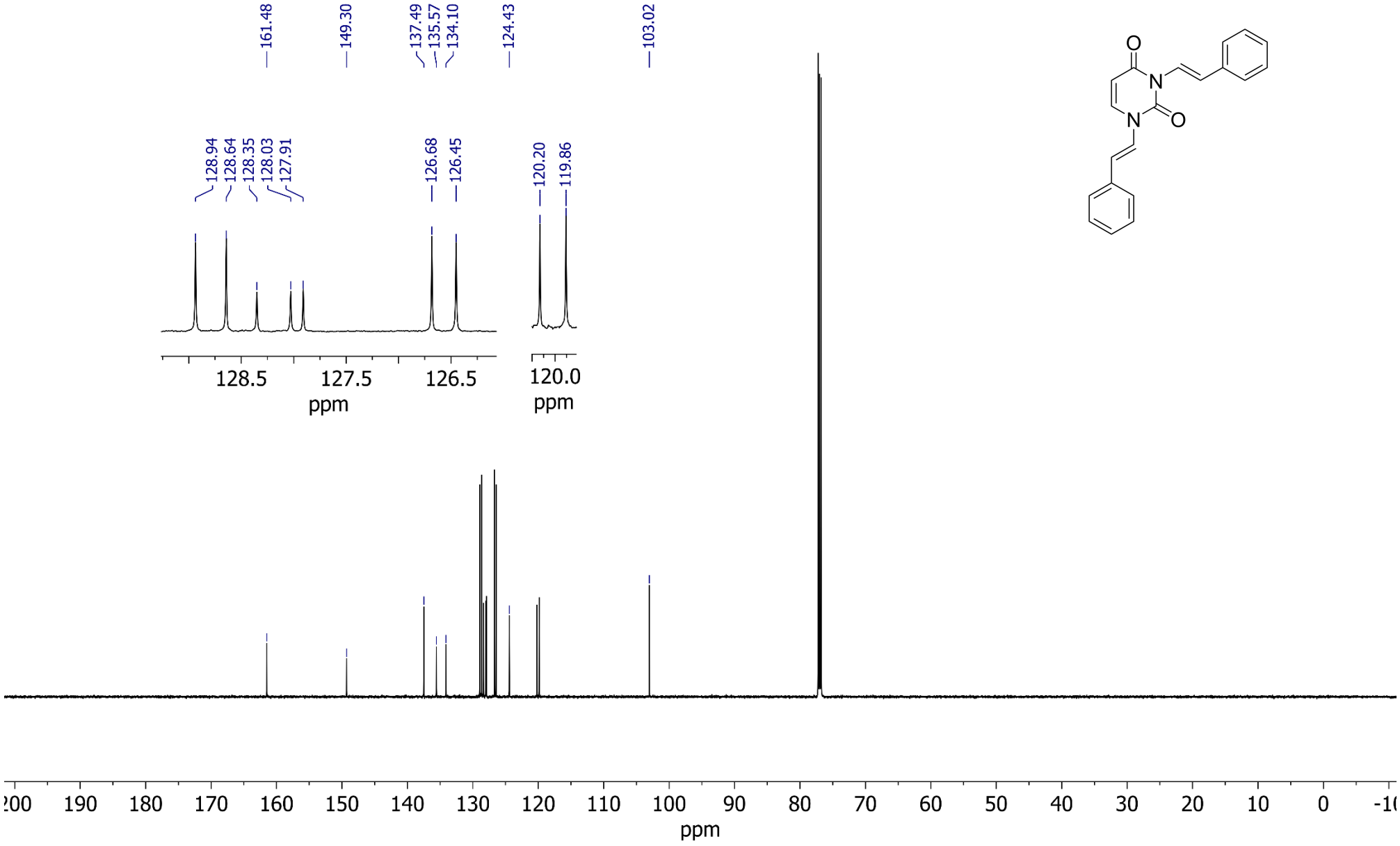
^{13}C NMR spectrum of 3p (151 MHz, $CDCl_3$)



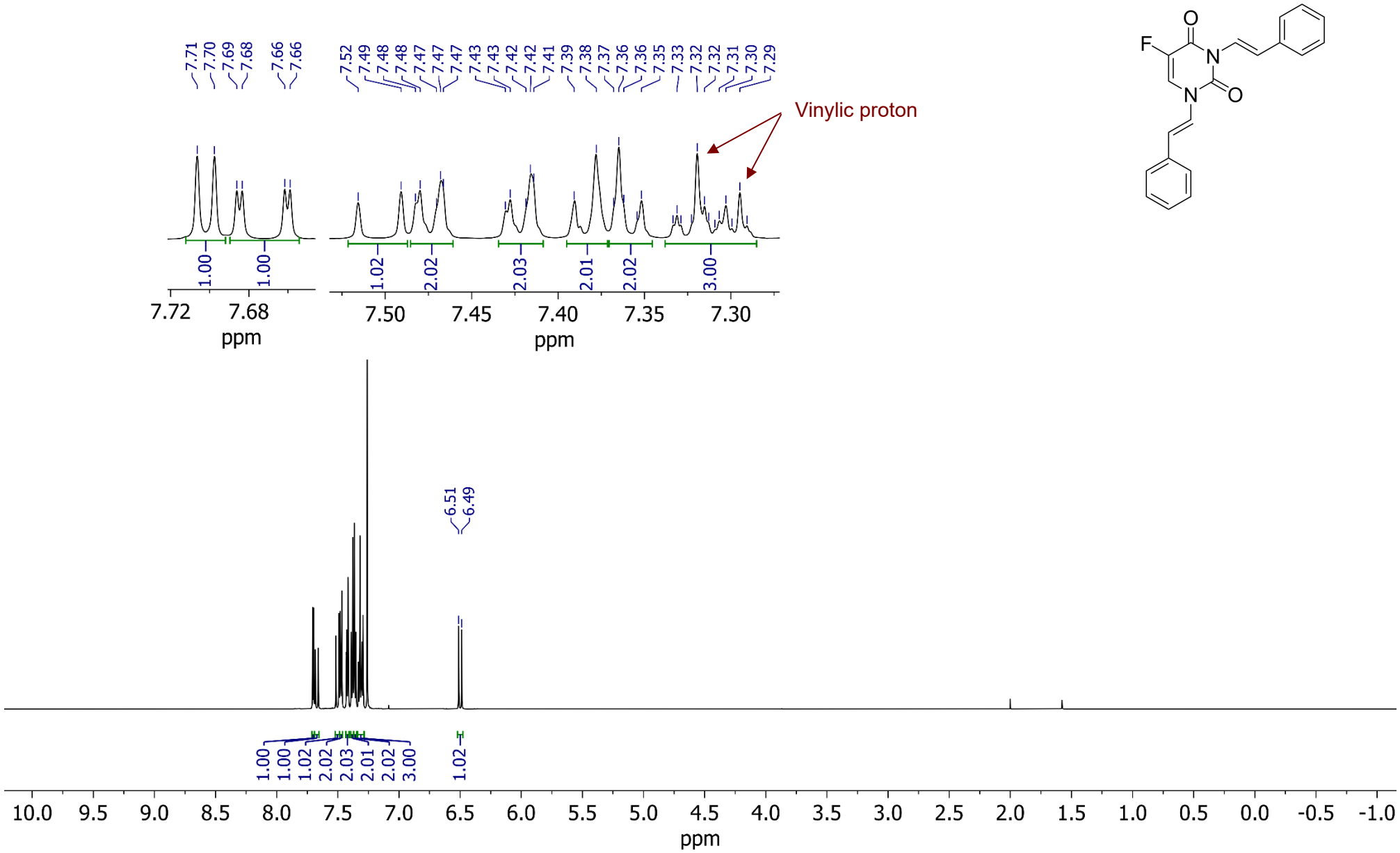
^{1}H NMR spectrum of 3q (600 MHz, $CDCl_3$)



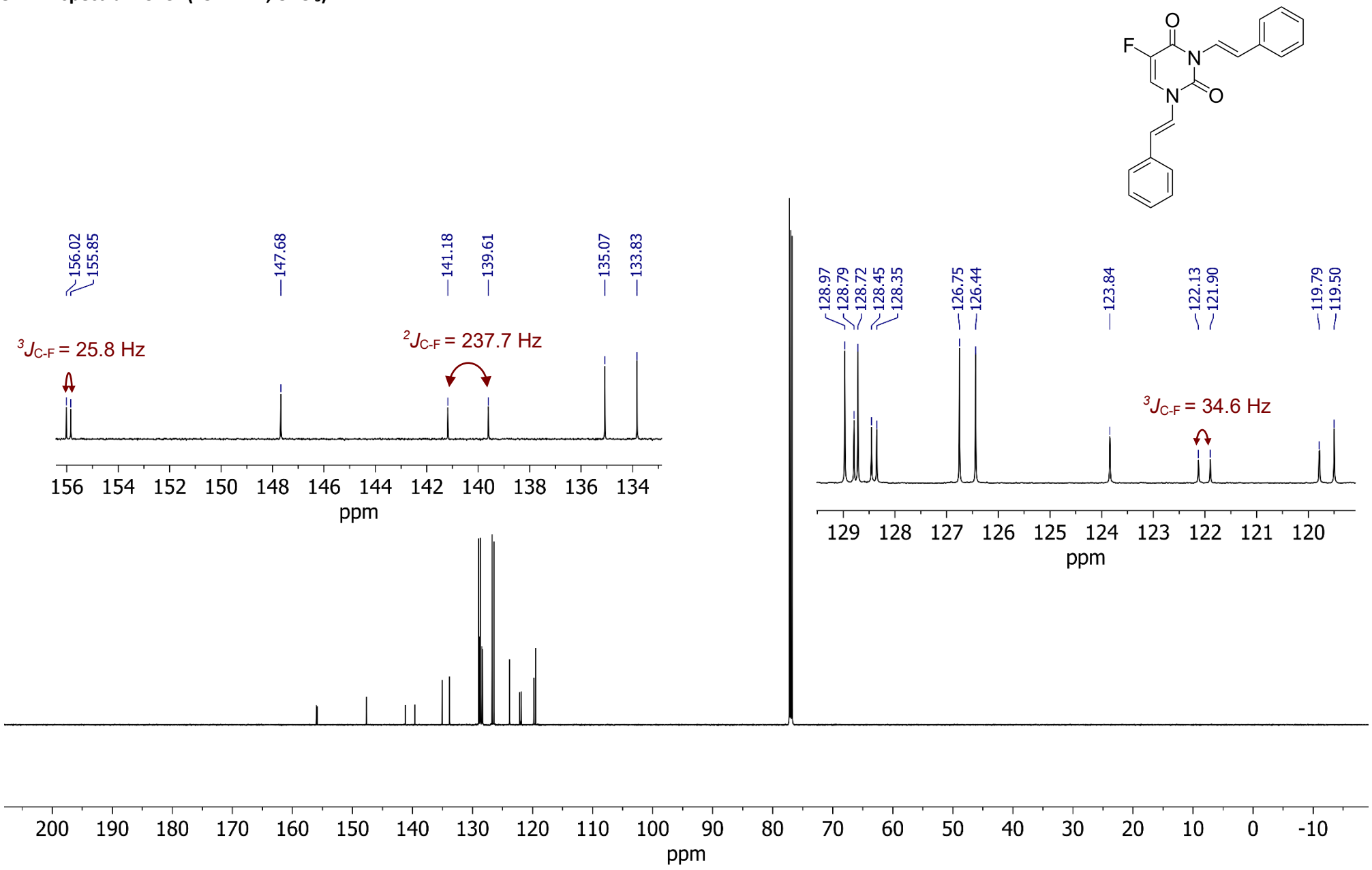
^{13}C NMR spectrum of 3q (151 MHz, $CDCl_3$)



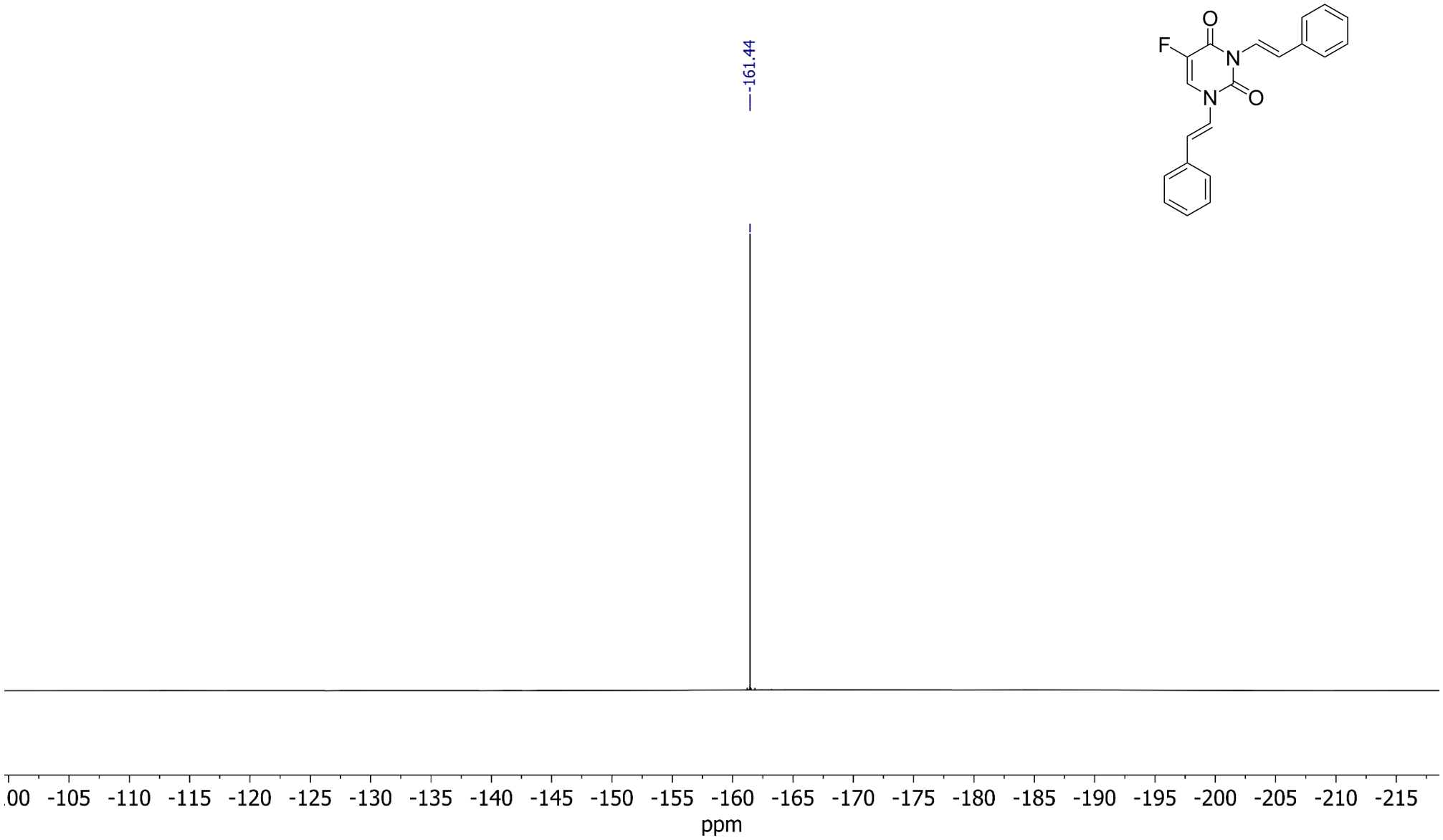
^{1}H NMR spectrum of 3r (600 MHz, $CDCl_3$)



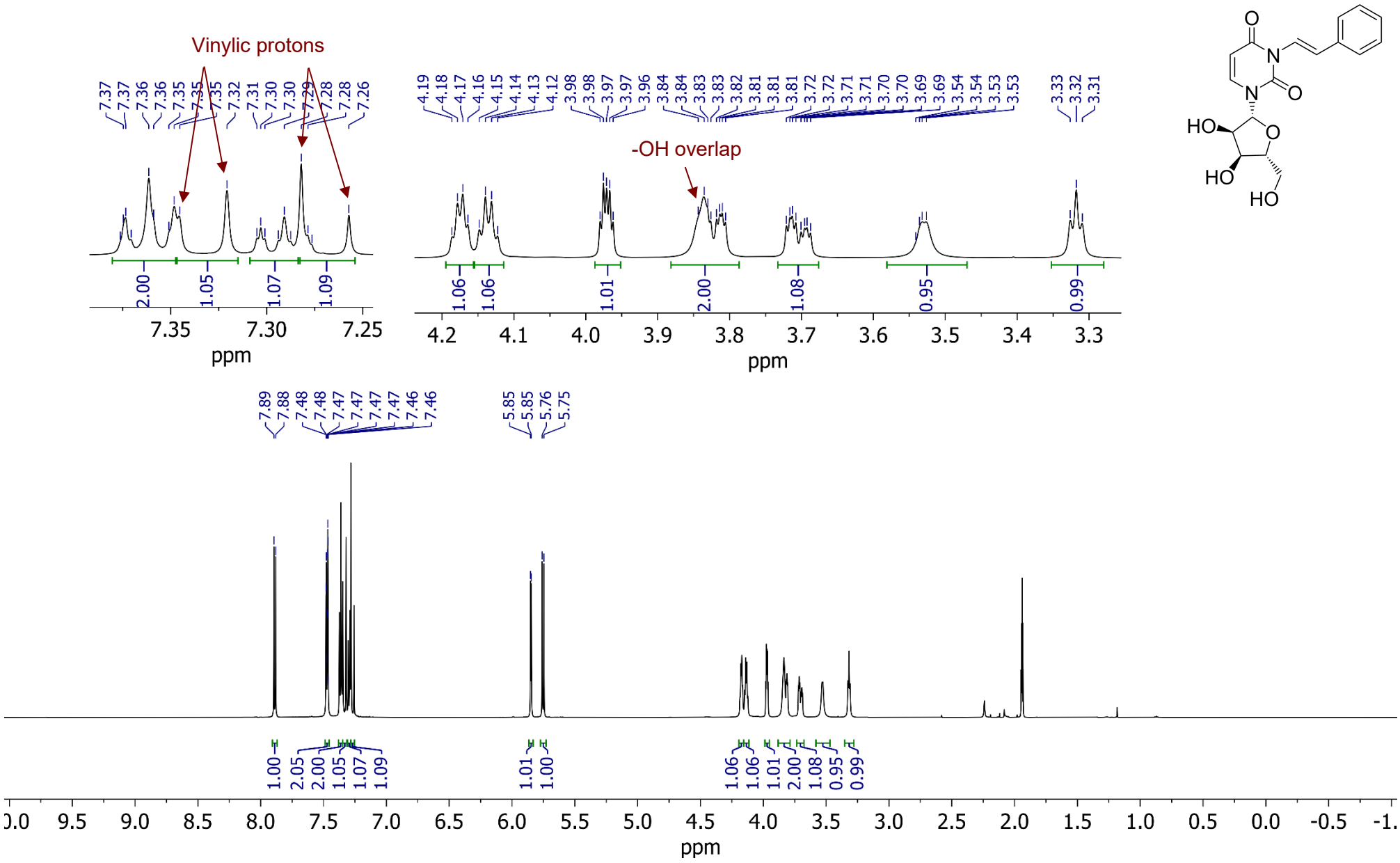
^{13}C NMR spectrum of 3r (151 MHz, $CDCl_3$)



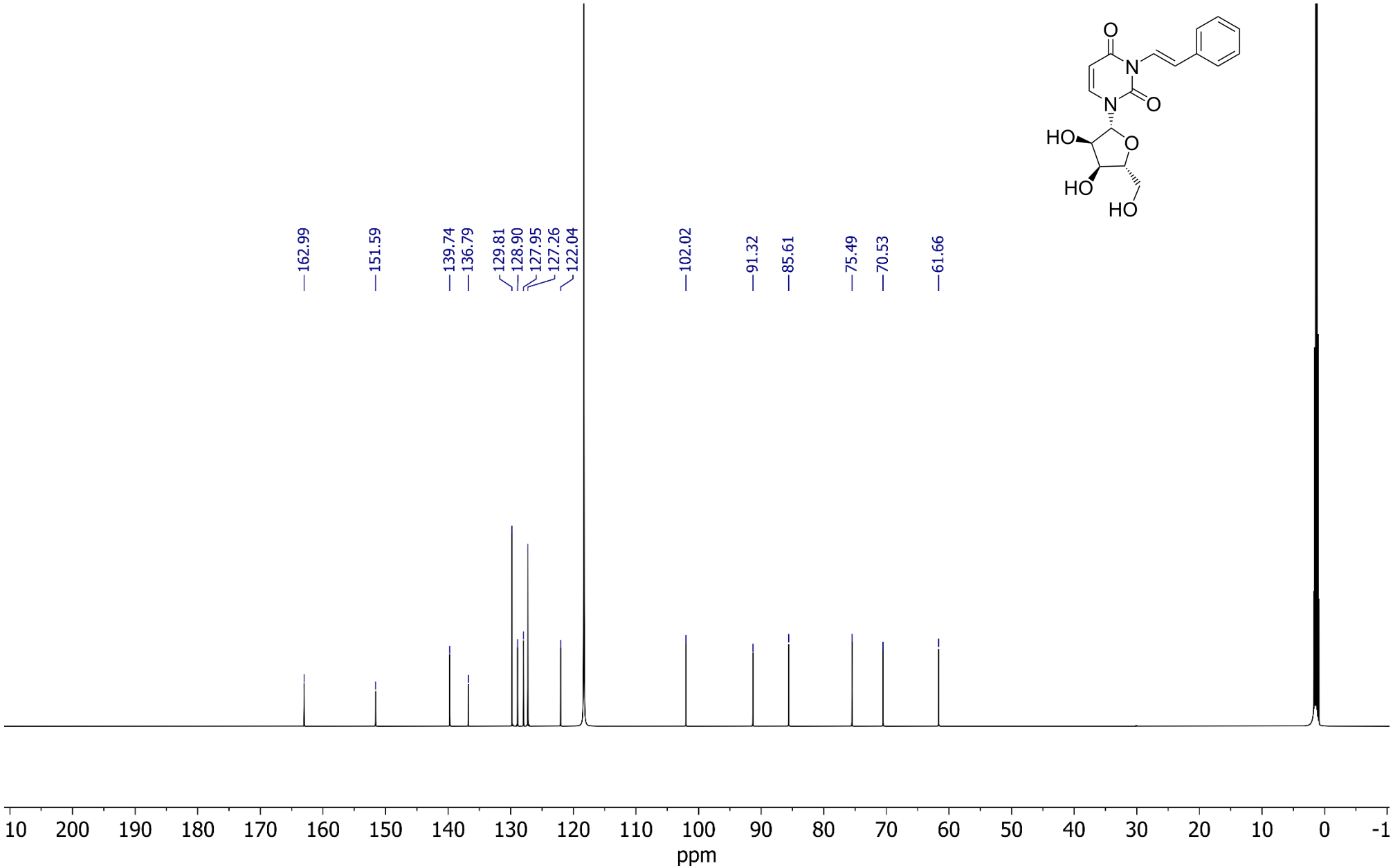
^{19}F NMR spectrum of 3r (376 MHz, $CDCl_3$)



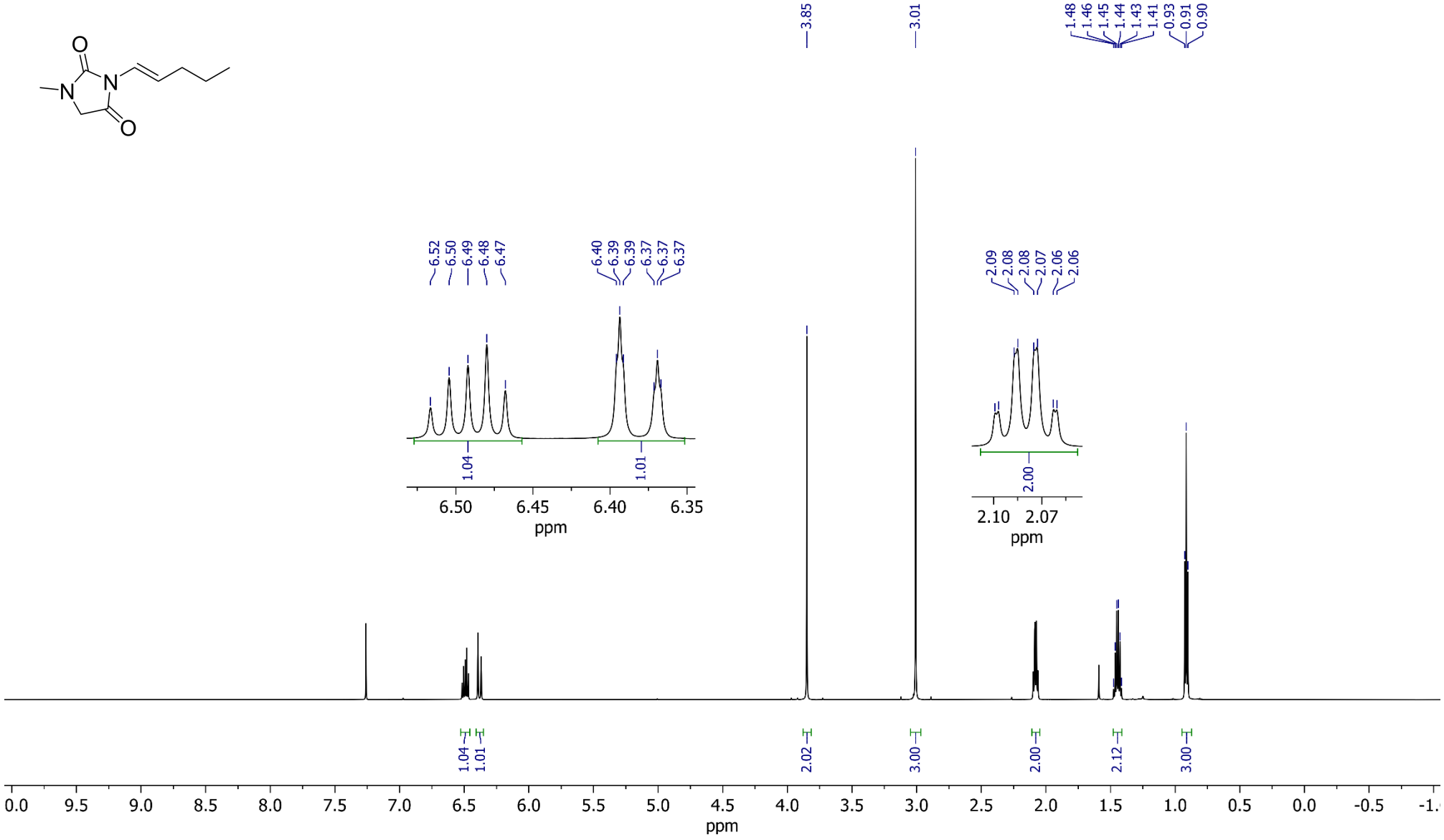
^{1}H NMR spectrum of 3s (600 MHz, CD_3CN)



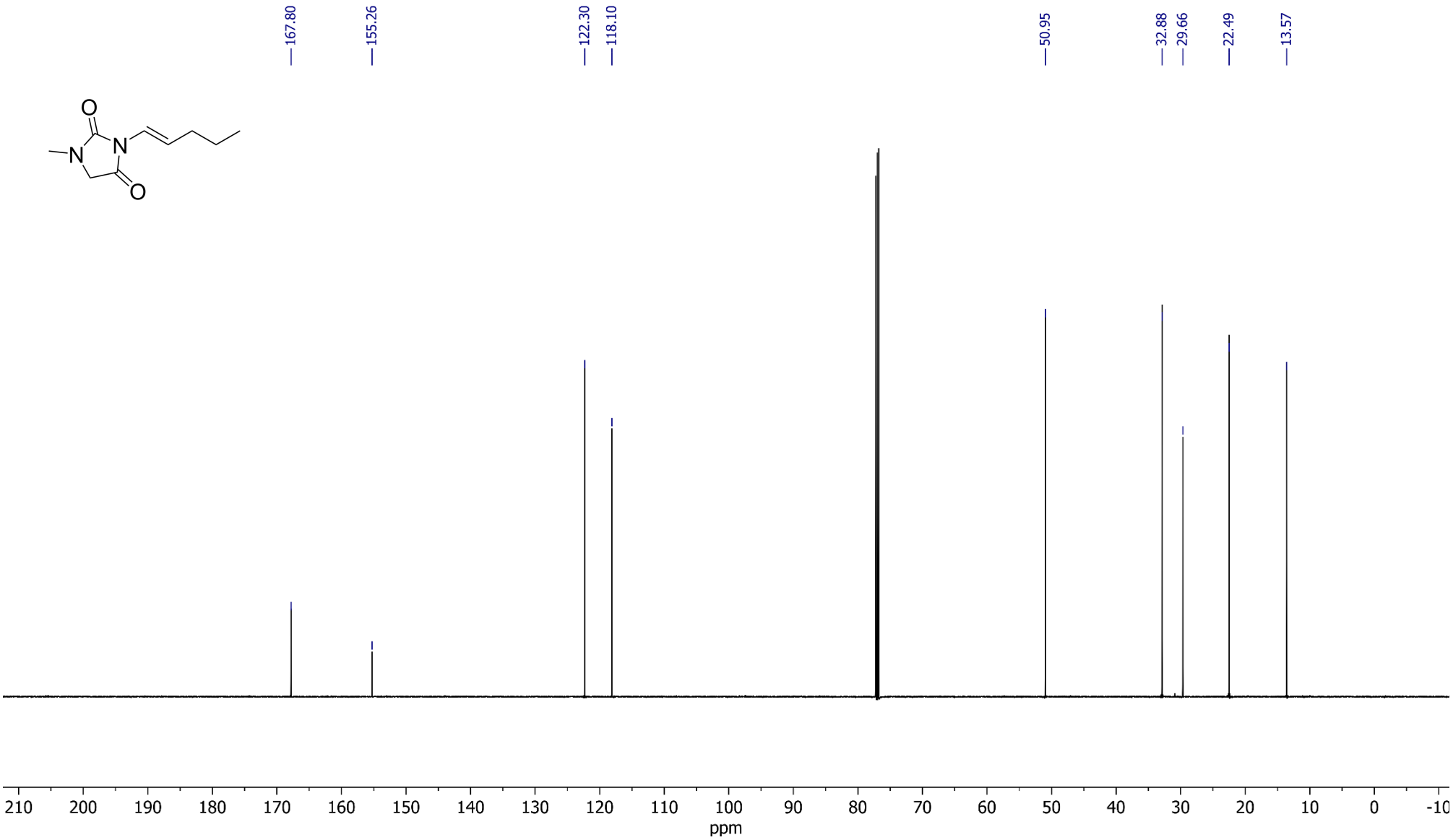
^{13}C NMR spectrum of 3s (151 MHz, CD_3CN)



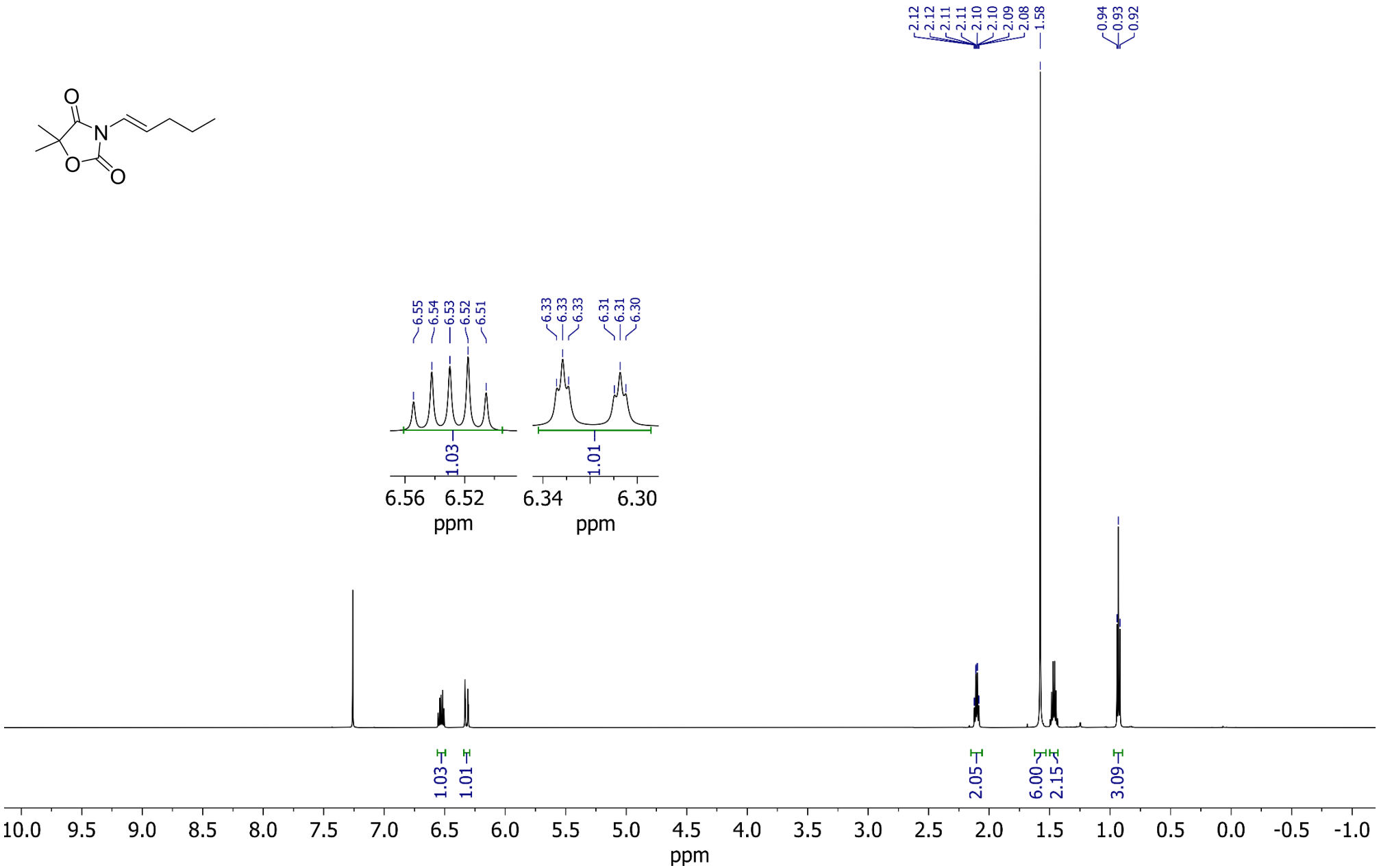
^{1}H NMR spectrum of 4a (600 MHz, $CDCl_3$)



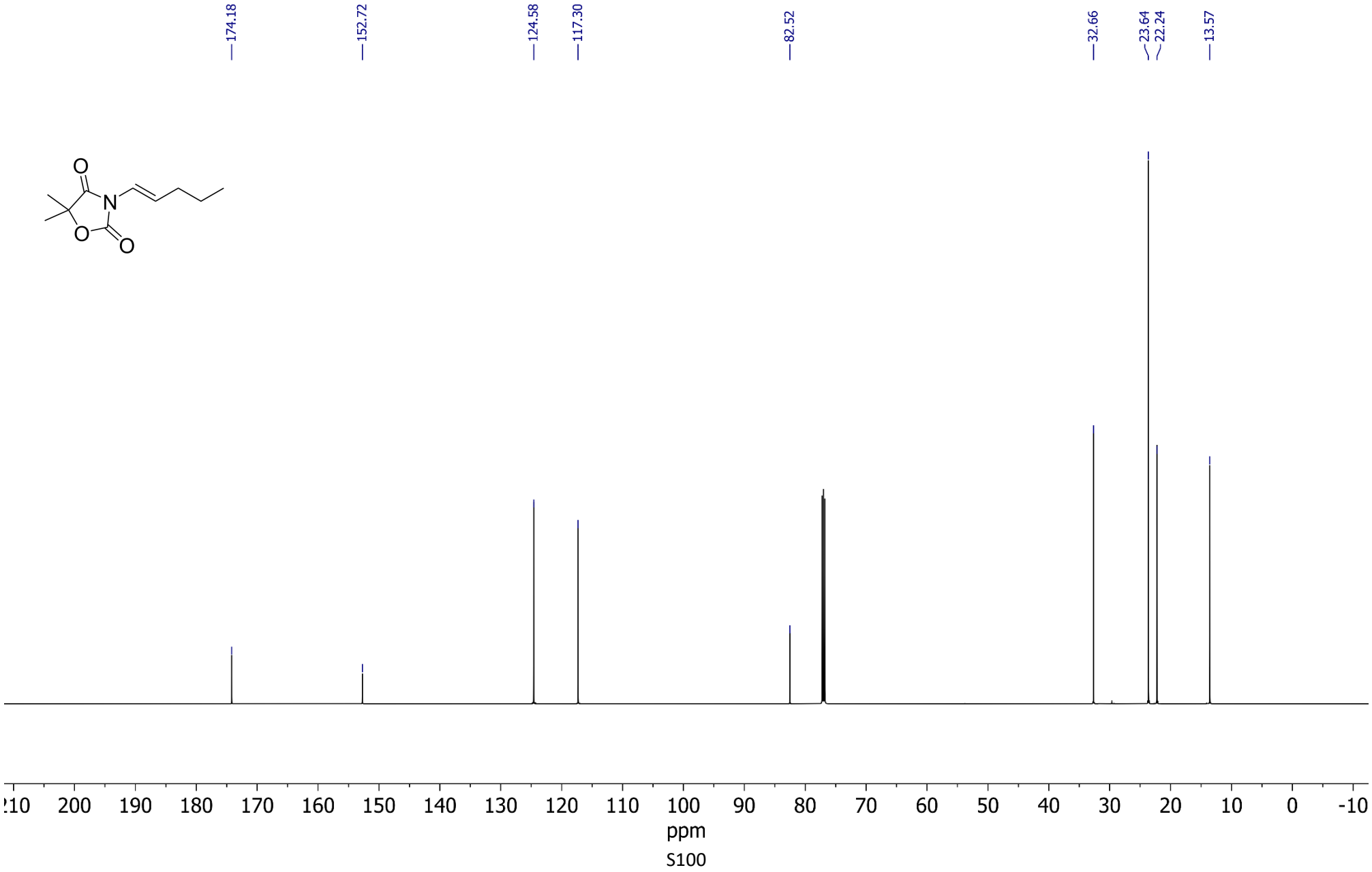
^{13}C NMR spectrum of 4a (151 MHz, $CDCl_3$)



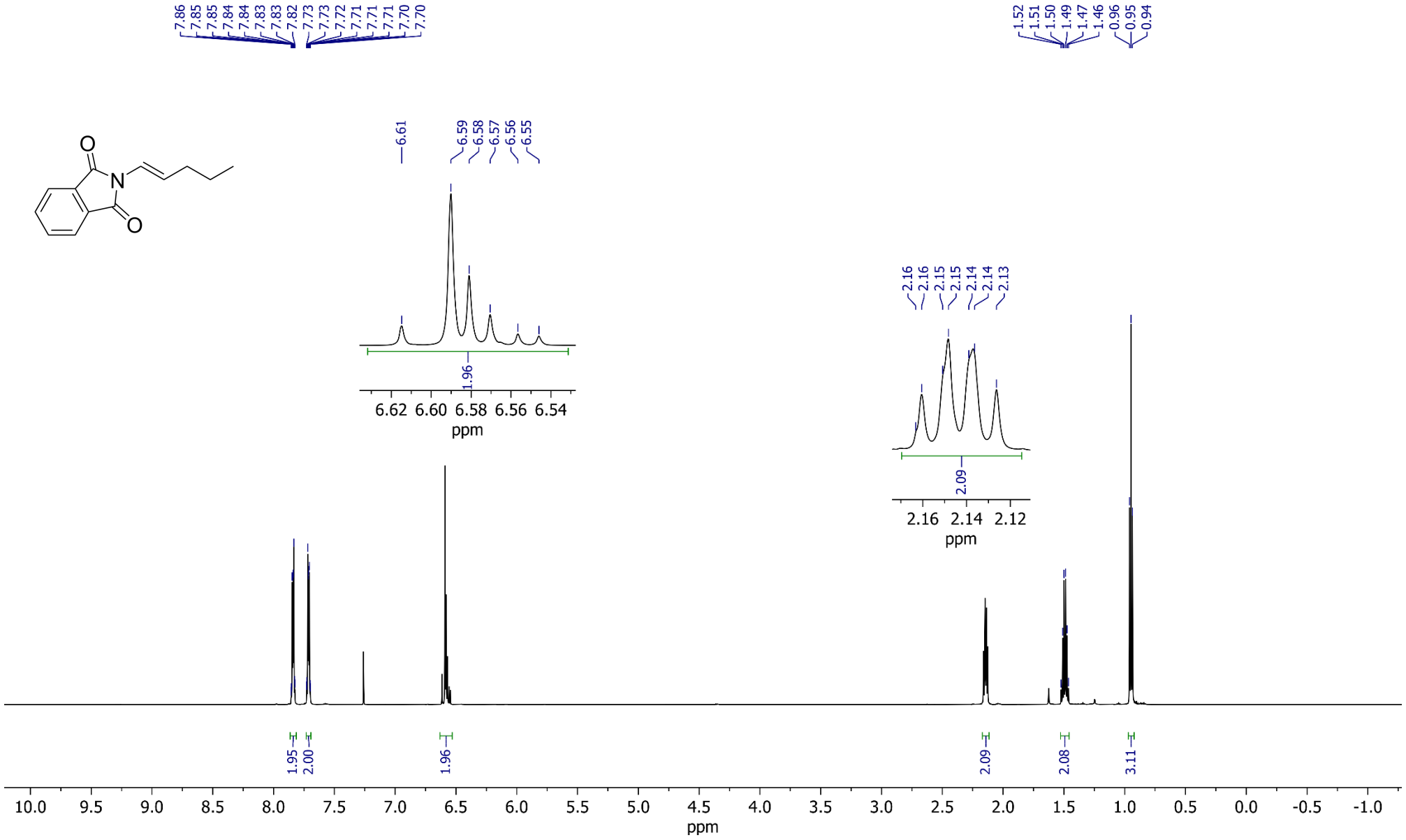
^{1}H NMR spectrum of 4b (600 MHz, $CDCl_3$)



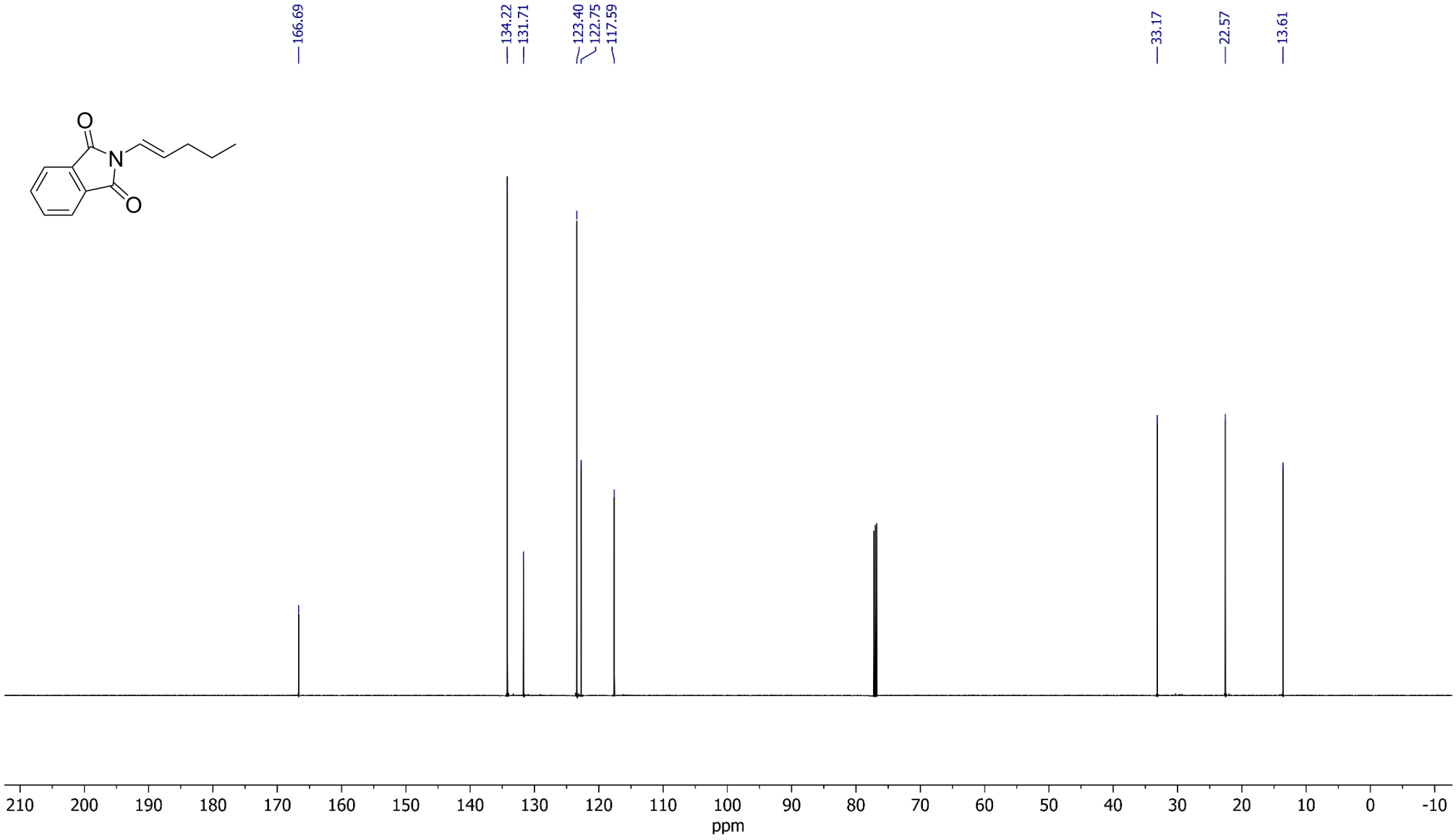
^{13}C NMR spectrum of 4b (151 MHz, $CDCl_3$)



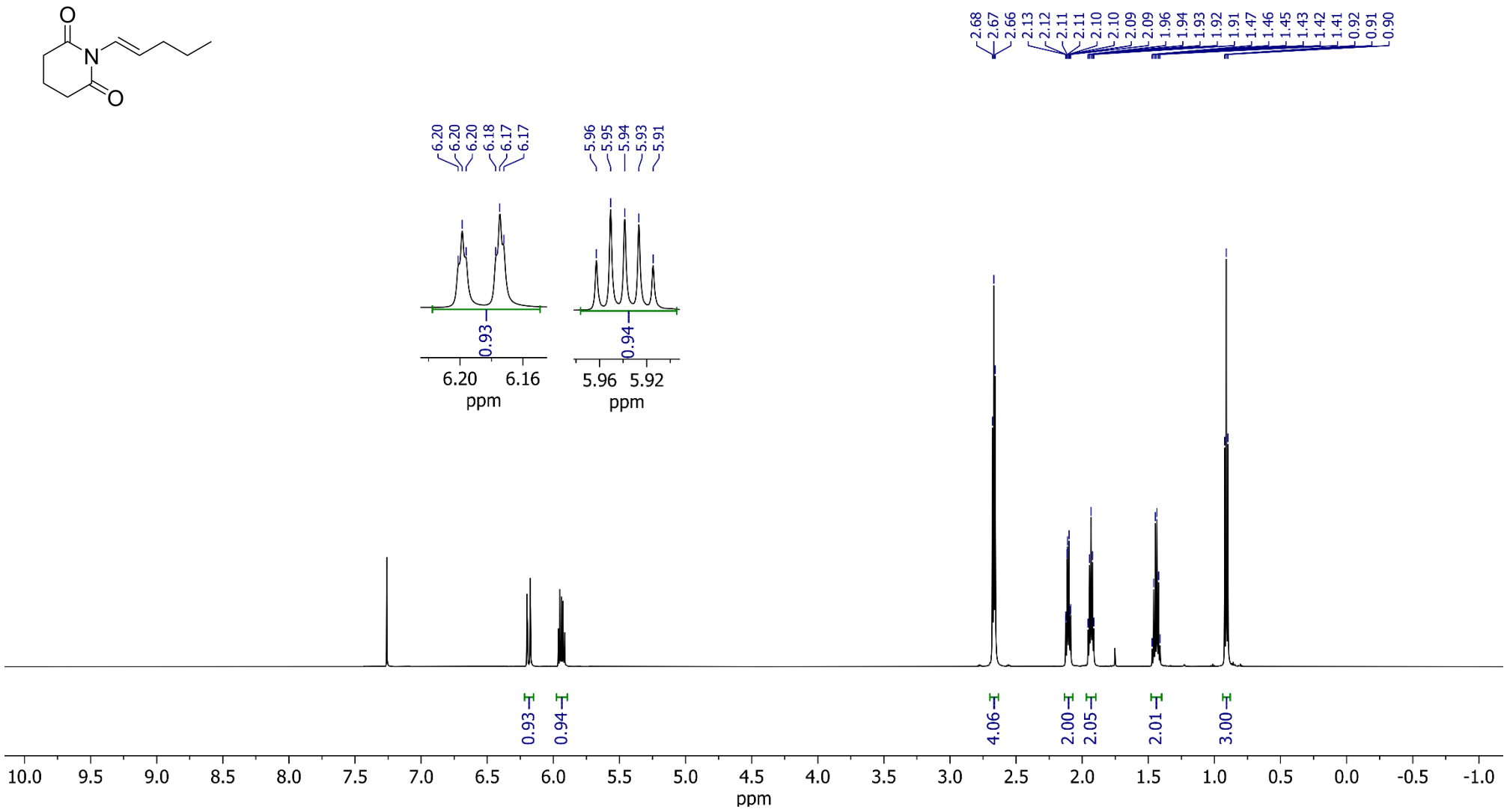
^{1}H NMR spectrum of 4c (600 MHz, $CDCl_3$)



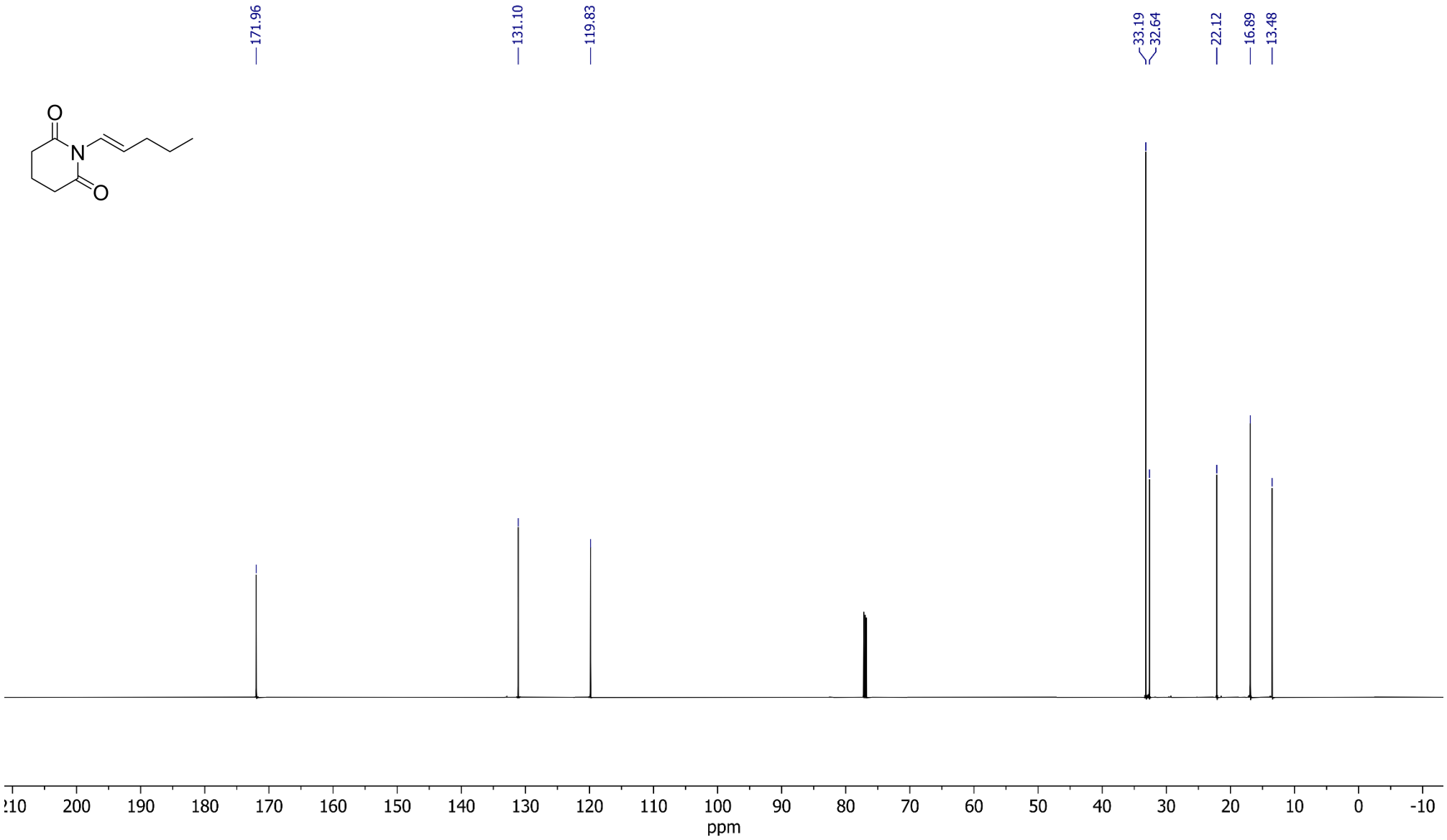
^{13}C NMR spectrum of 4c (151 MHz, $CDCl_3$)



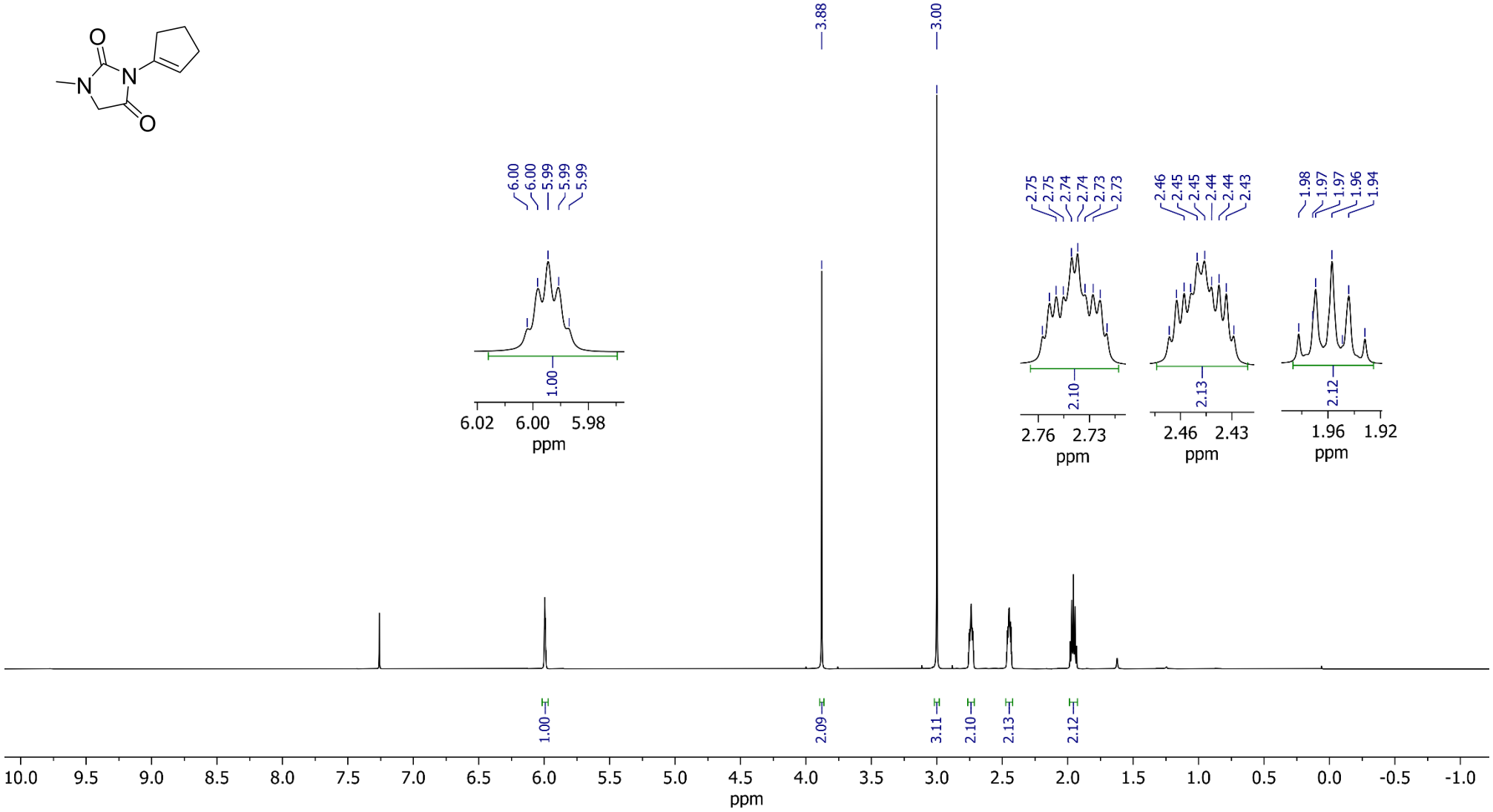
^{1}H NMR spectrum of 4d (600 MHz, $CDCl_3$)



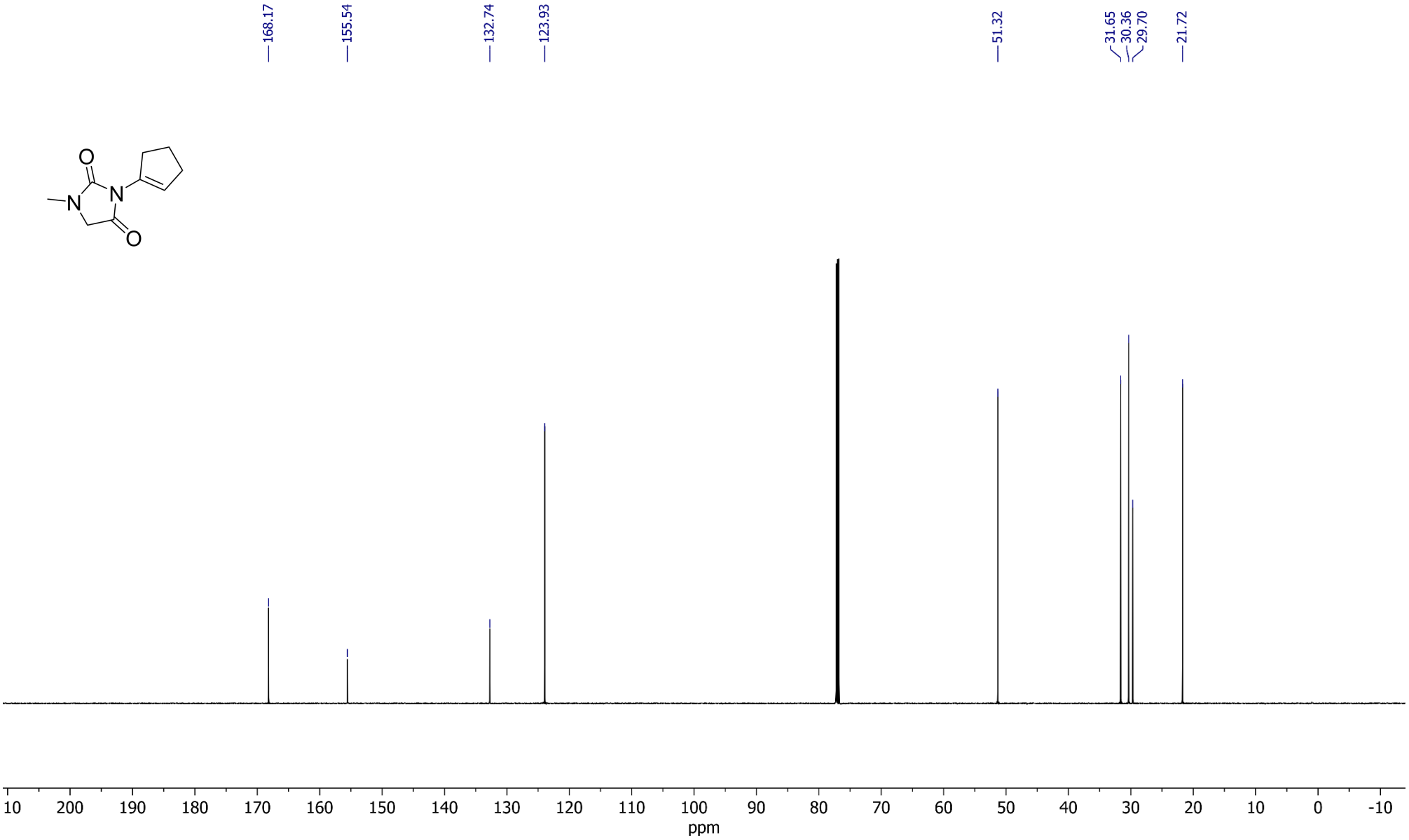
^{13}C NMR spectrum of 4d (151 MHz, $CDCl_3$)



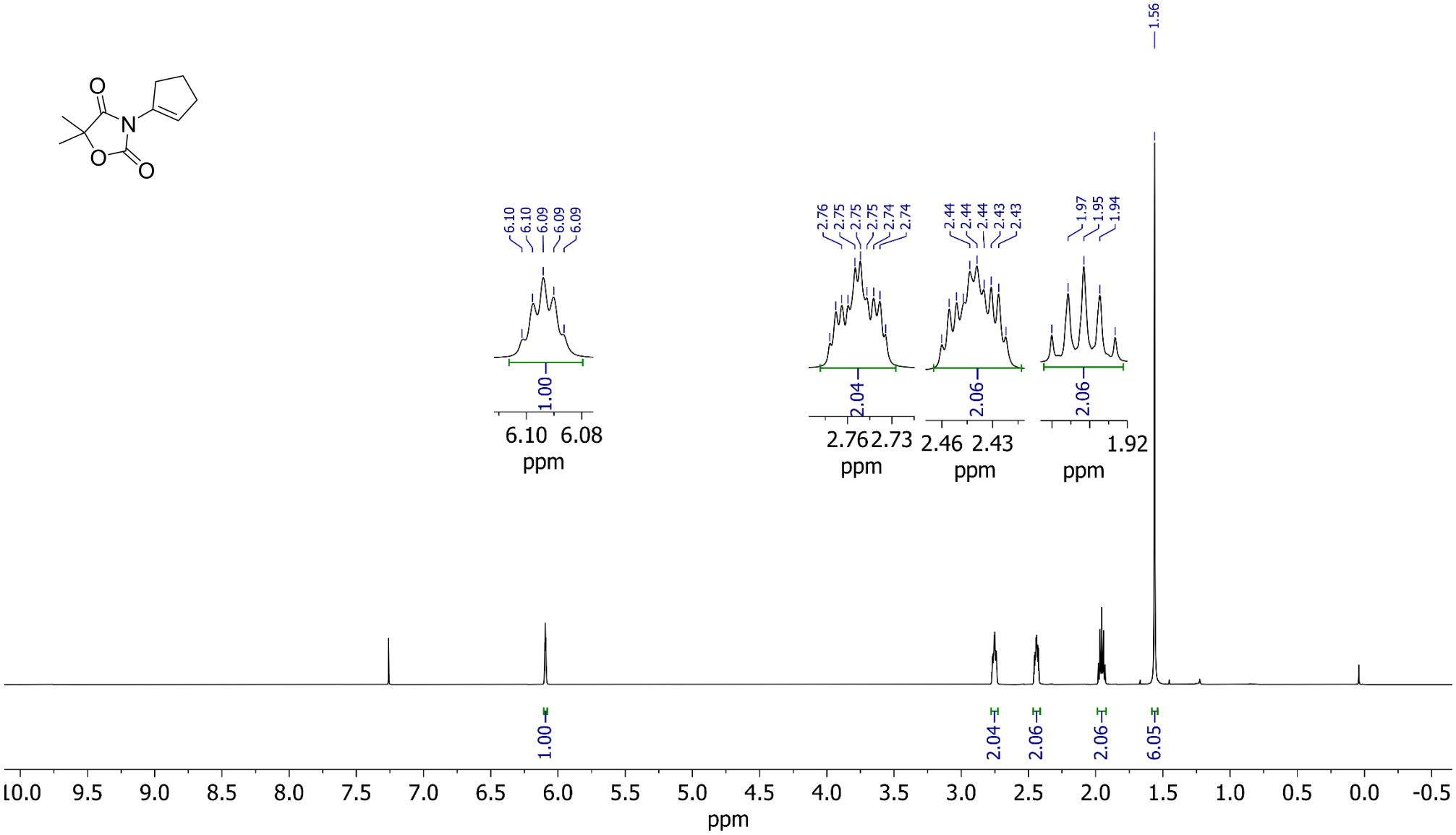
^{1}H NMR spectrum of 4e (600 MHz, $CDCl_3$)



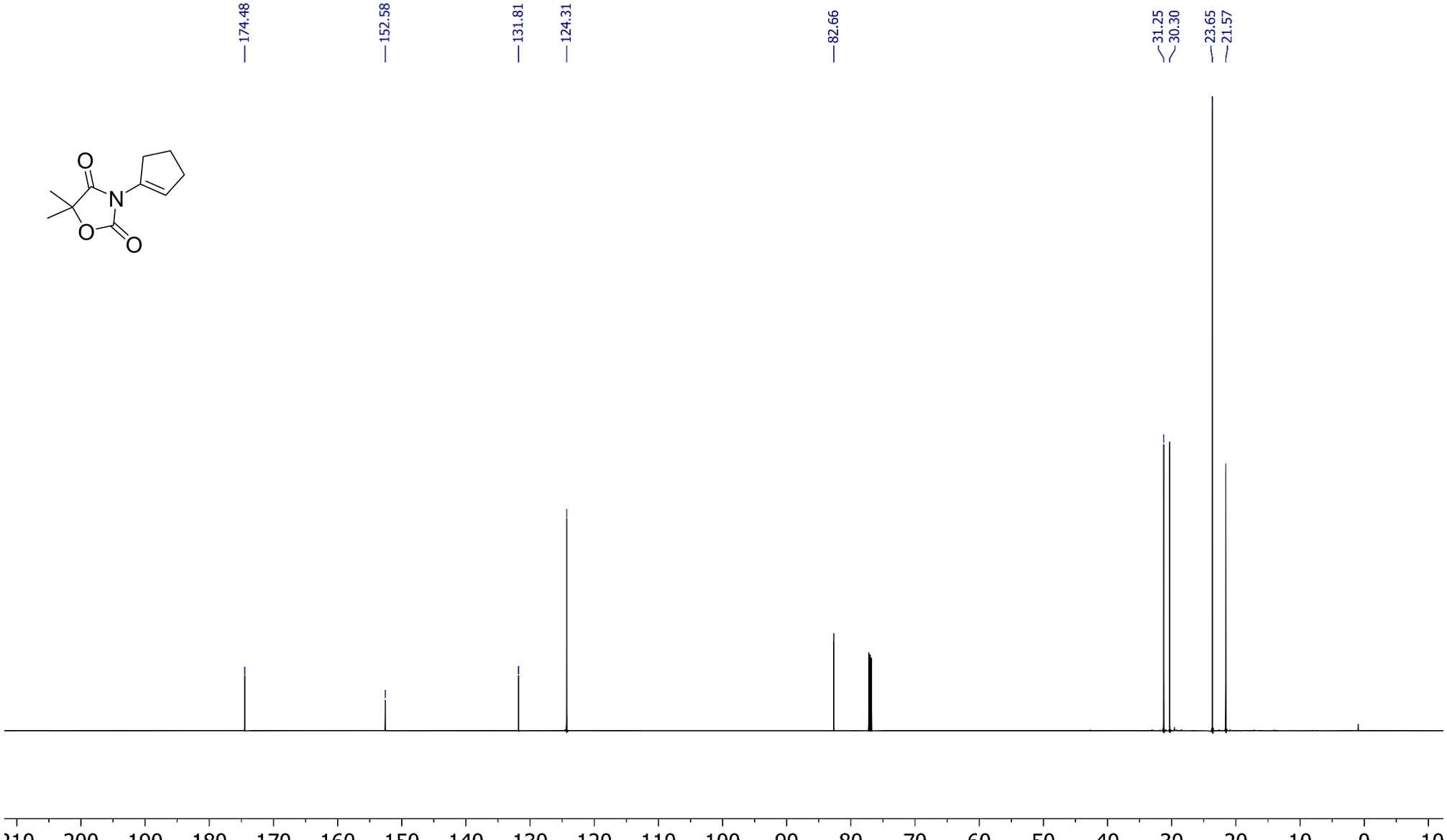
^{13}C NMR spectrum of 4e (151 MHz, $CDCl_3$)



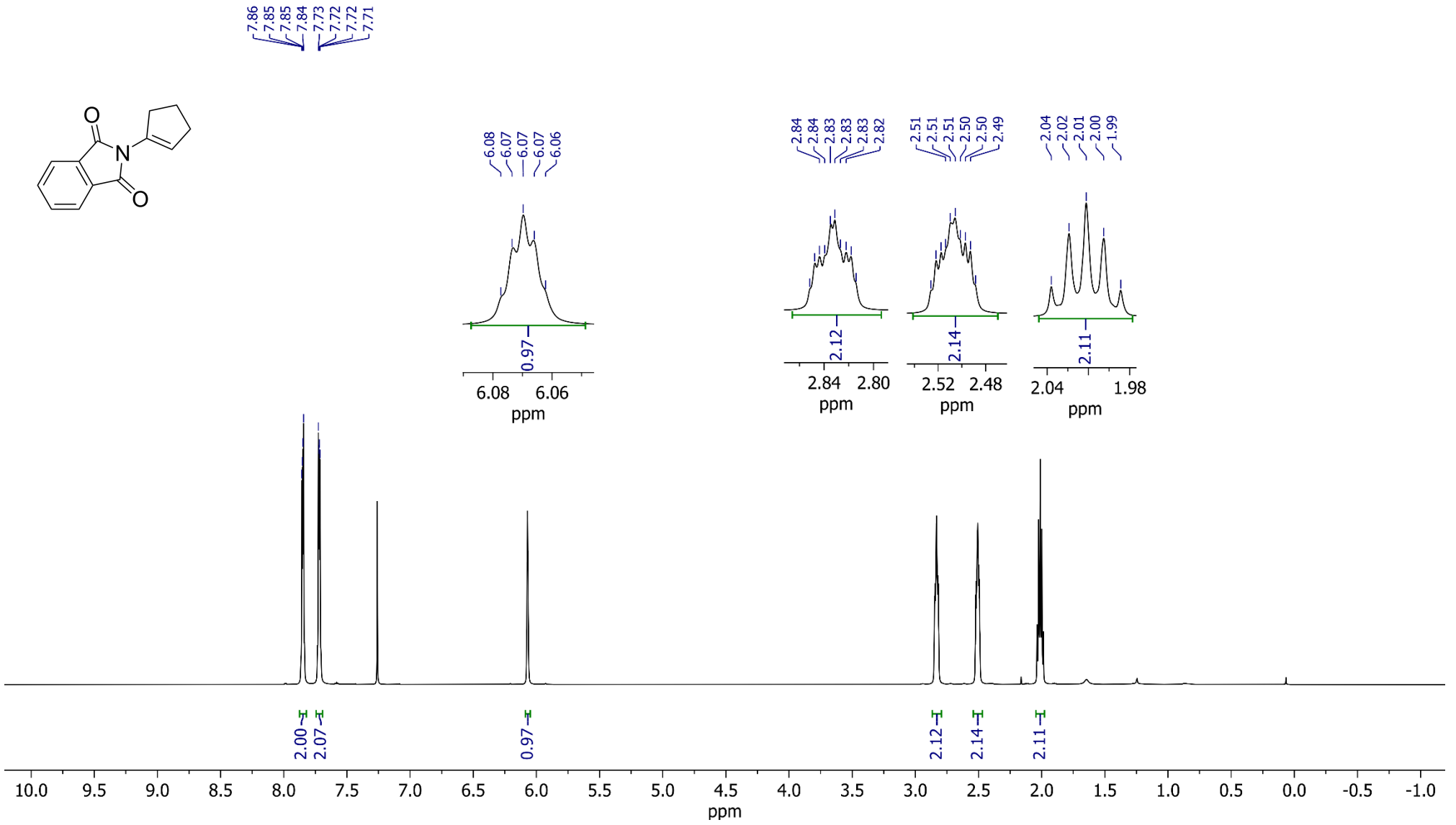
^{1}H NMR spectrum of 4g (600 MHz, $CDCl_3$)



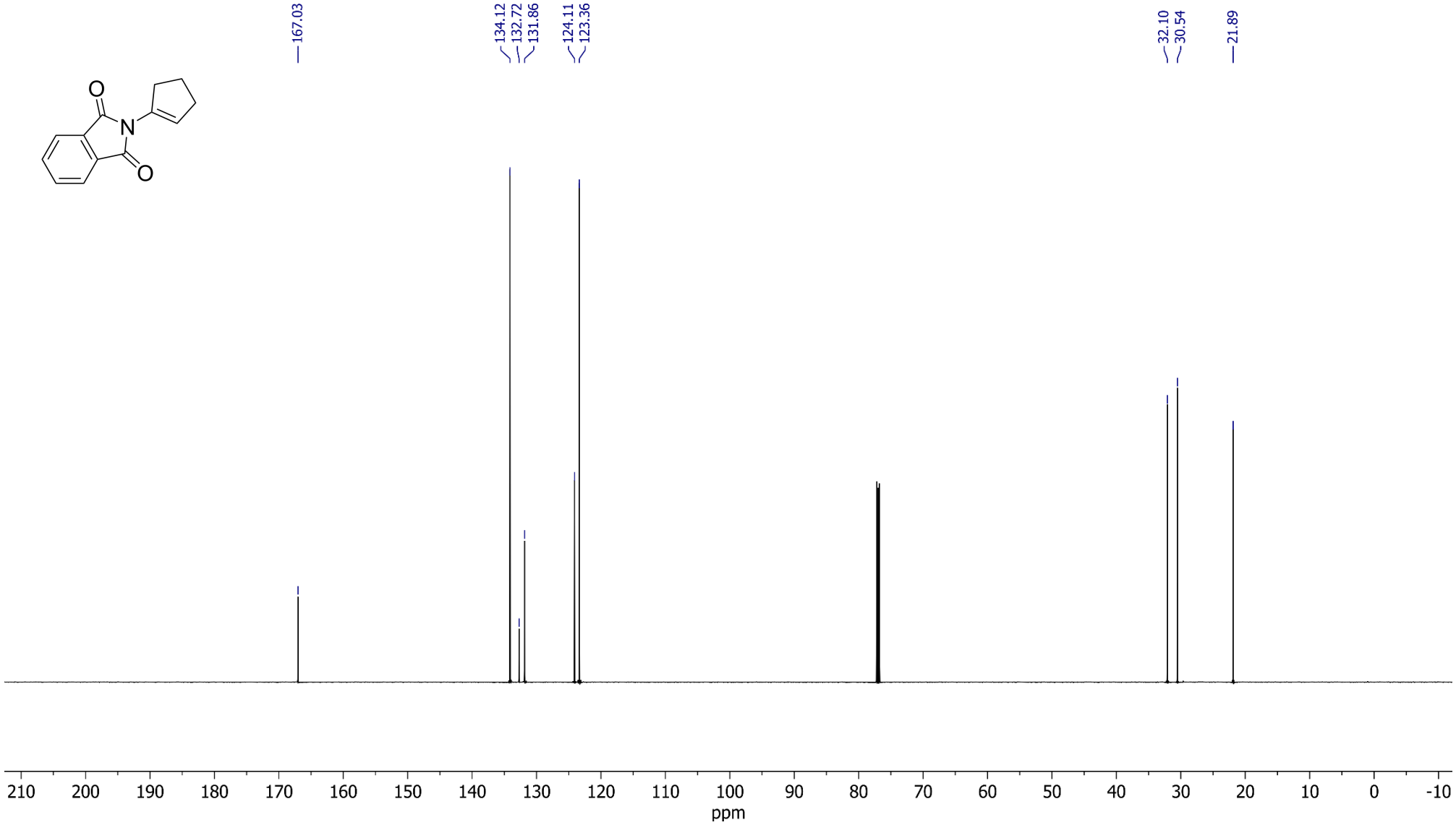
^{13}C NMR spectrum of 4g (151 MHz, $CDCl_3$)



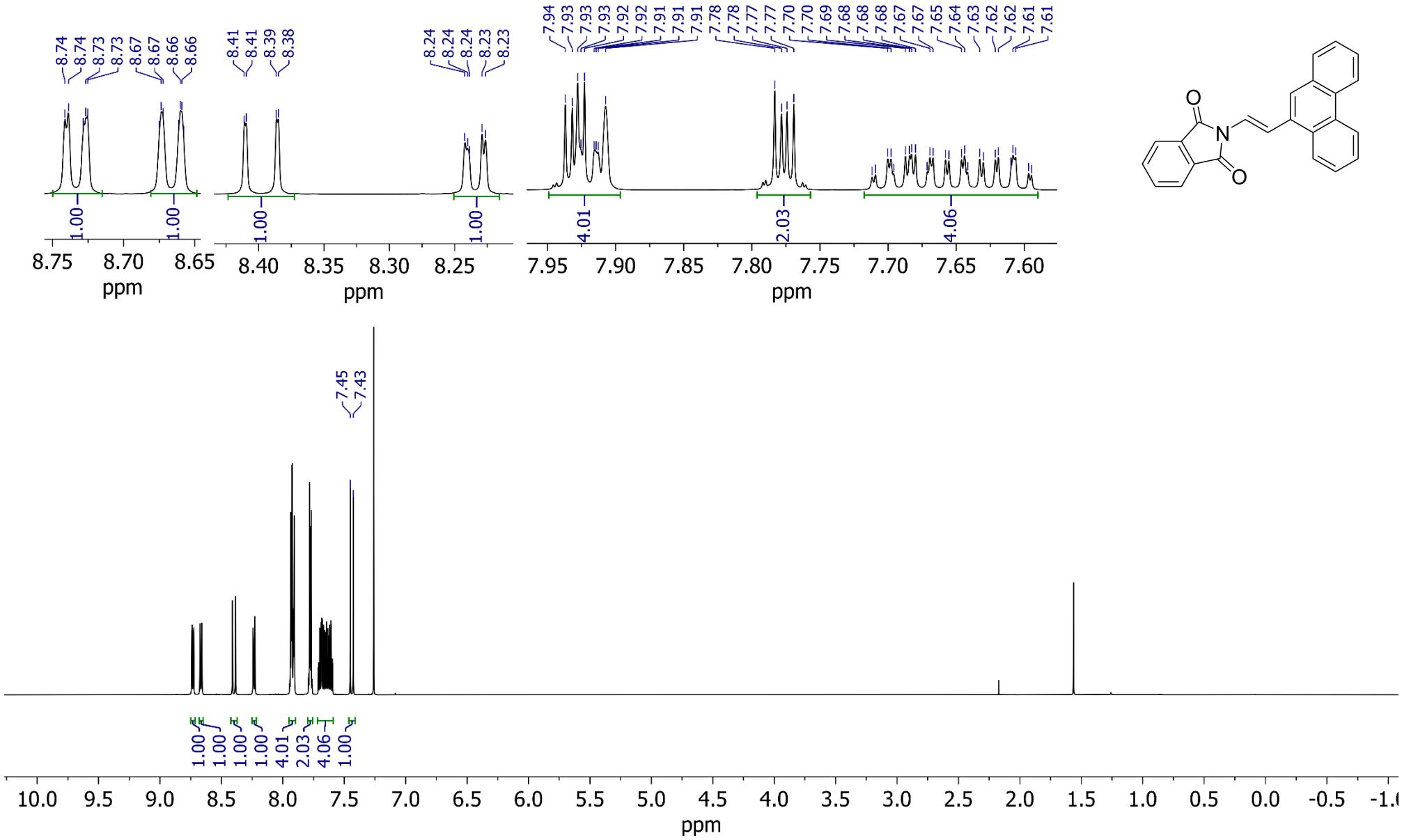
^{1}H NMR spectrum of 4h (600 MHz, $CDCl_3$)



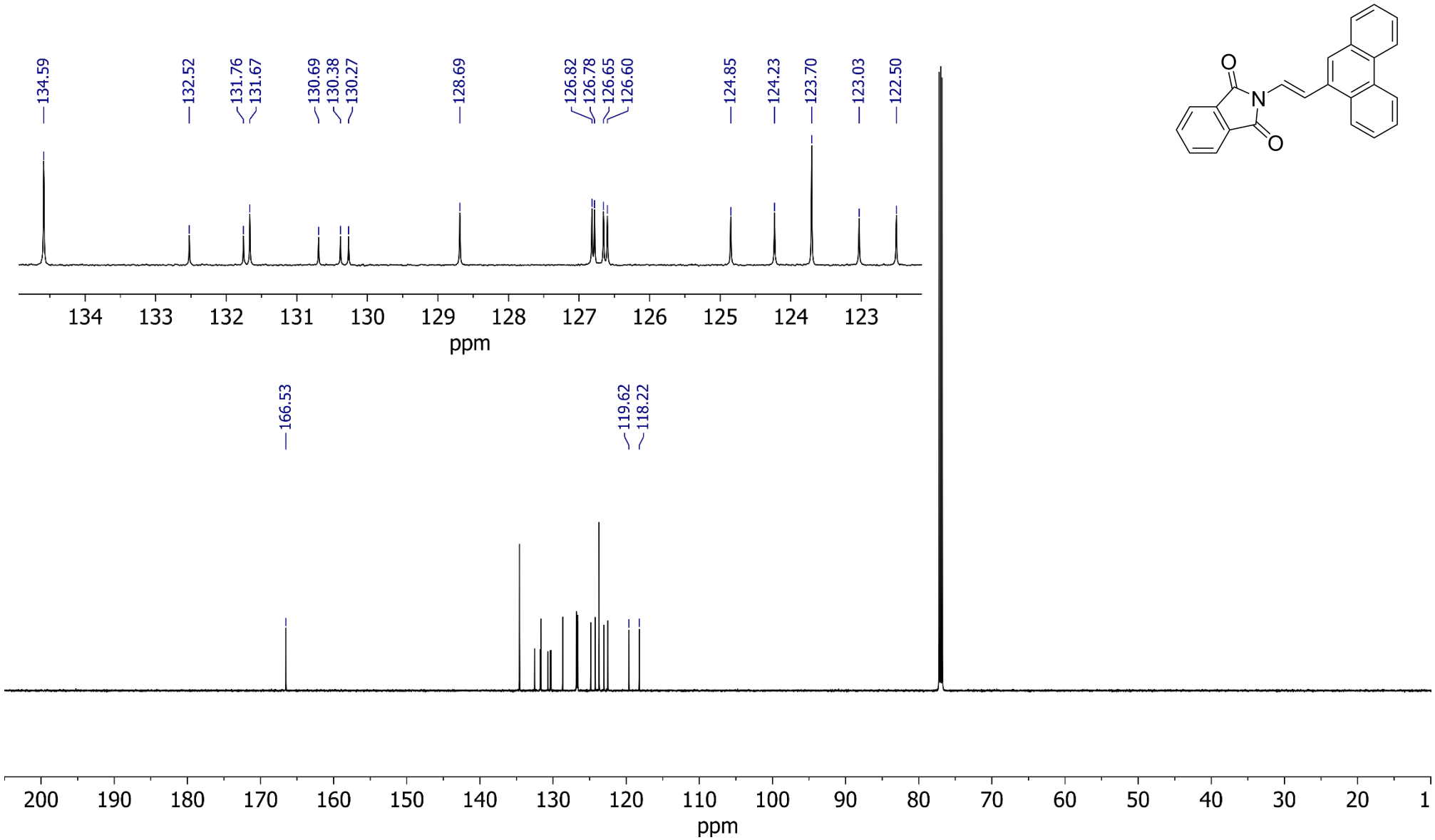
^{13}C NMR spectrum of 4h (151 MHz, $CDCl_3$)



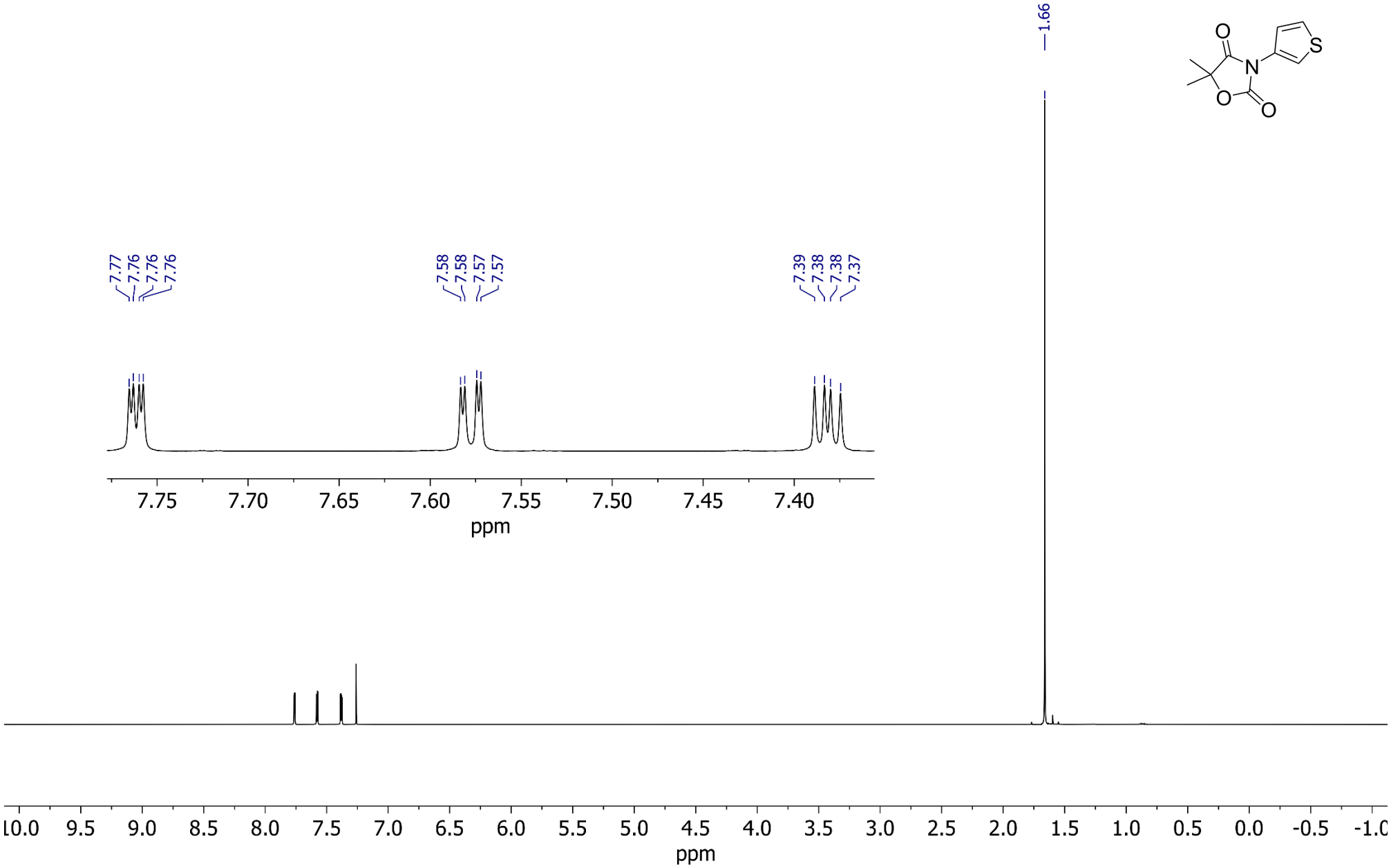
^{1}H NMR spectrum of 4i (600 MHz, $CDCl_3$)



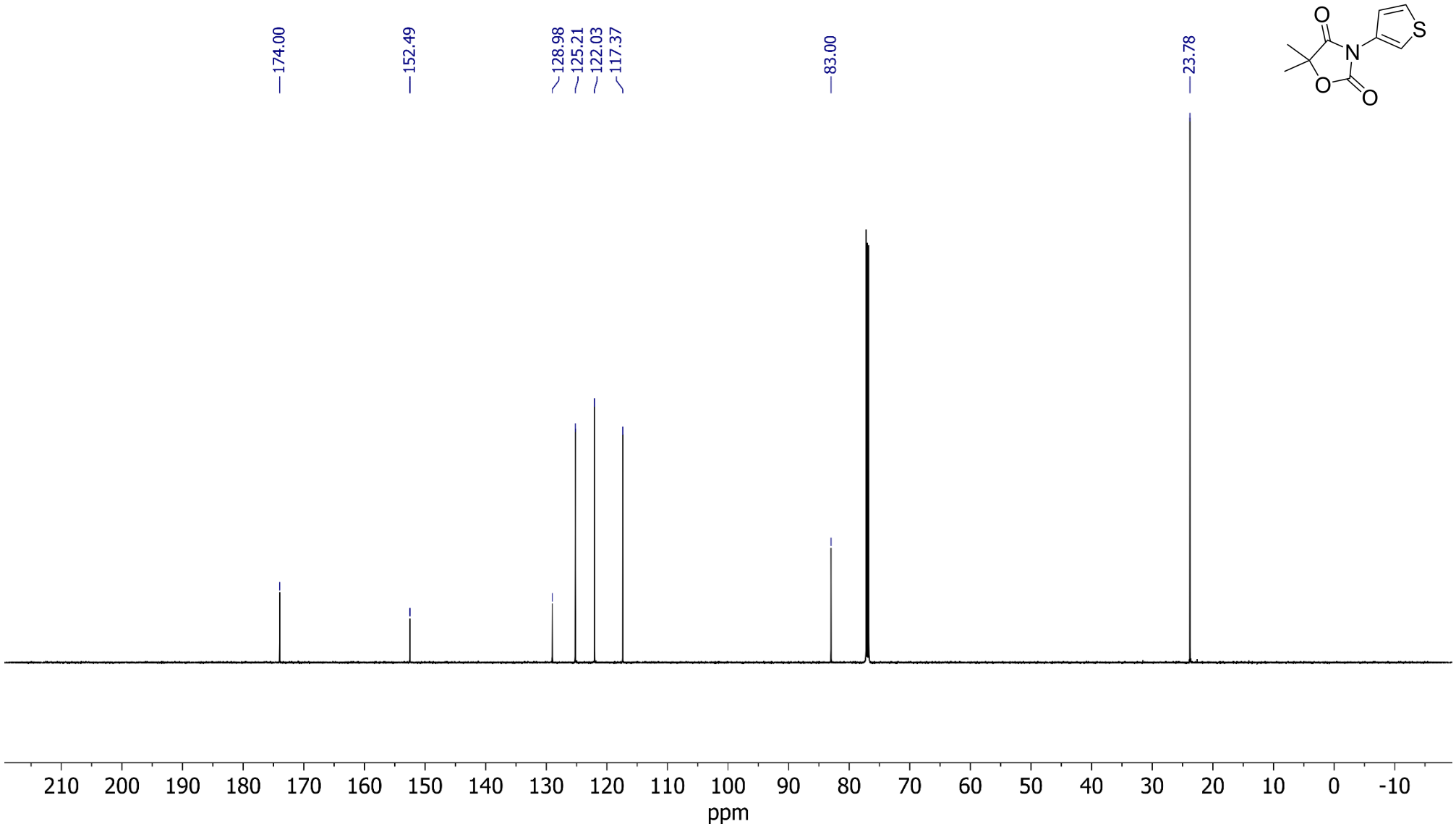
^{13}C NMR spectrum of 4i (151 MHz, $CDCl_3$)



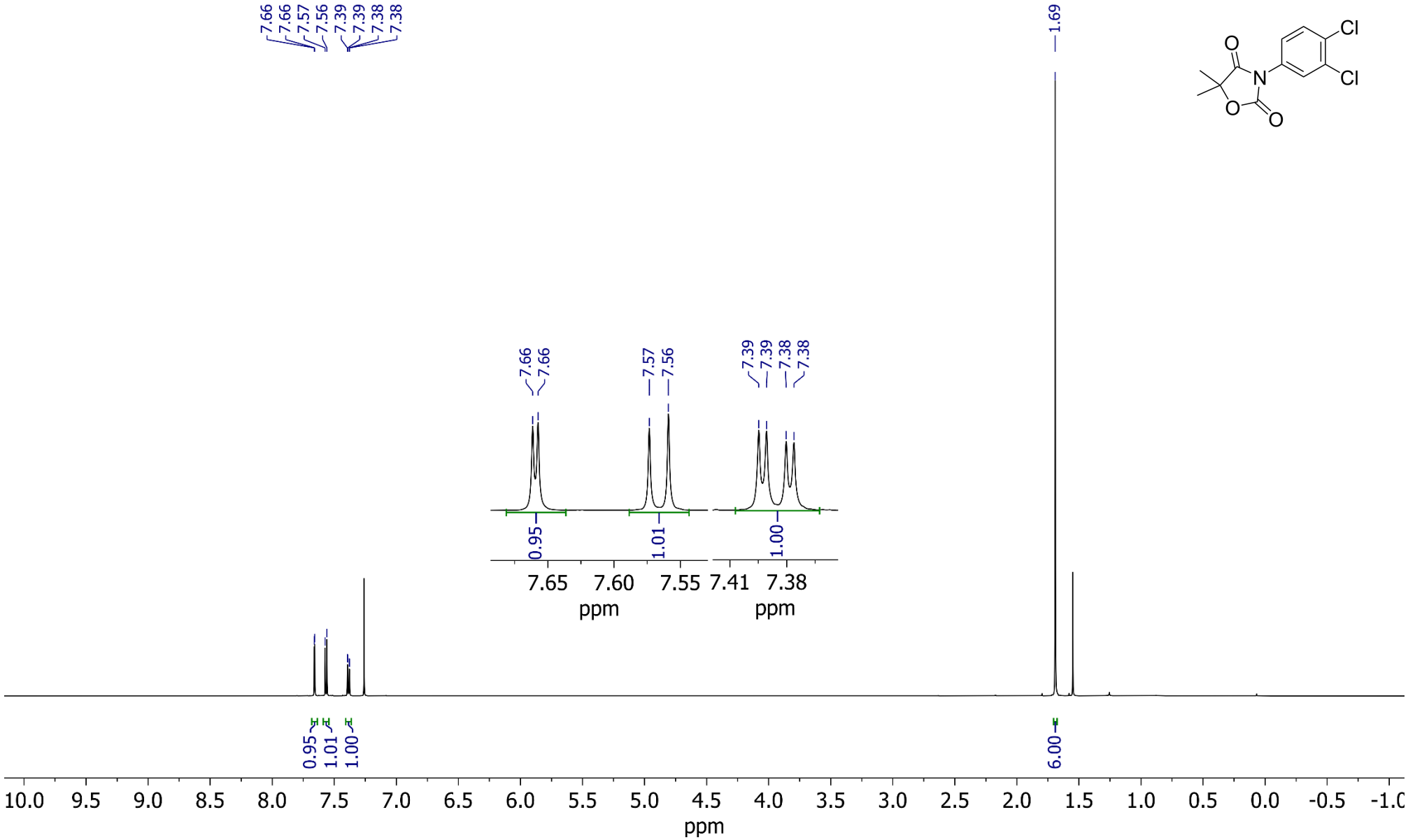
^{1}H NMR spectrum of 7a (600 MHz, $CDCl_3$)



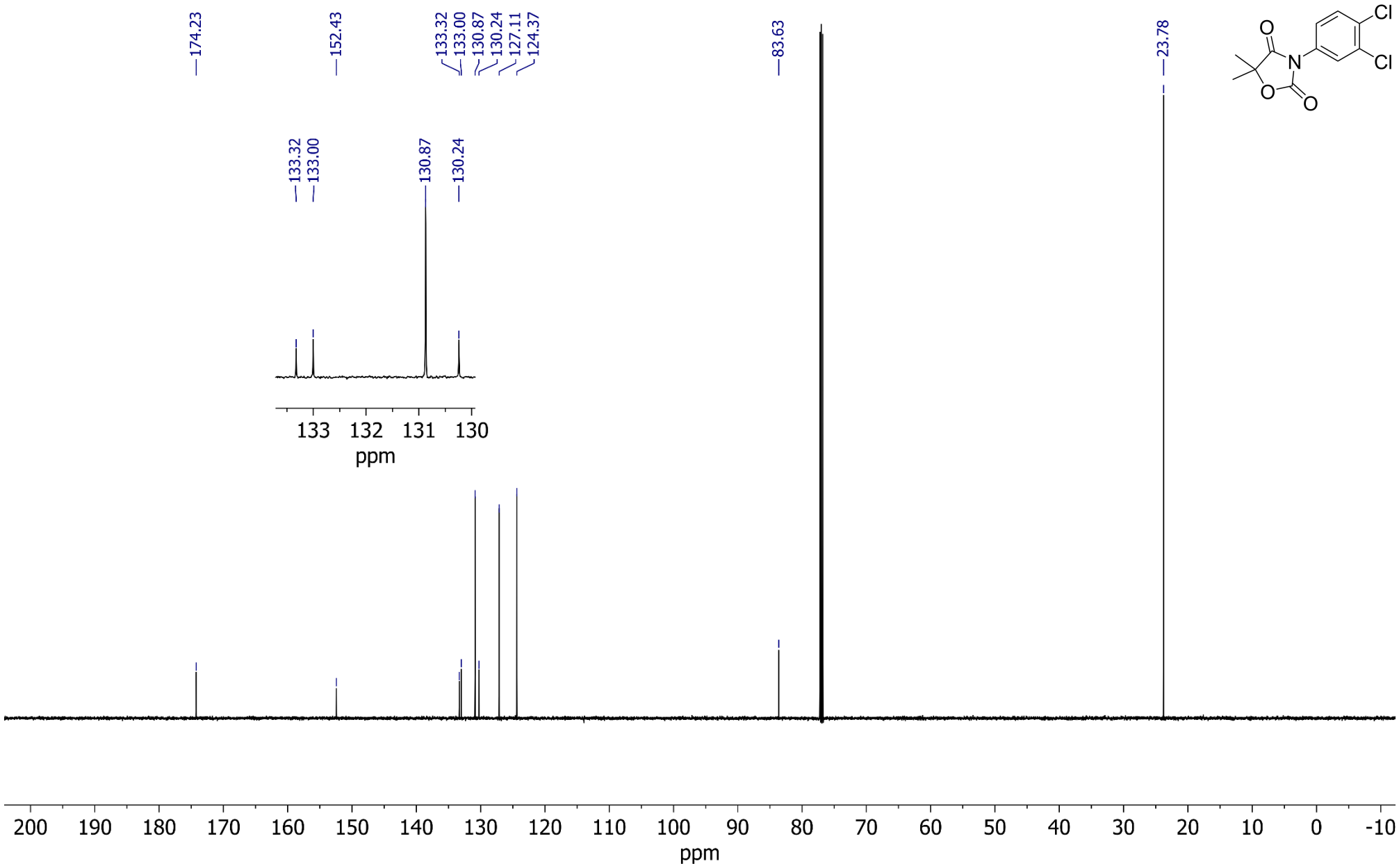
^{13}C NMR spectrum of 7a (151 MHz, $CDCl_3$)



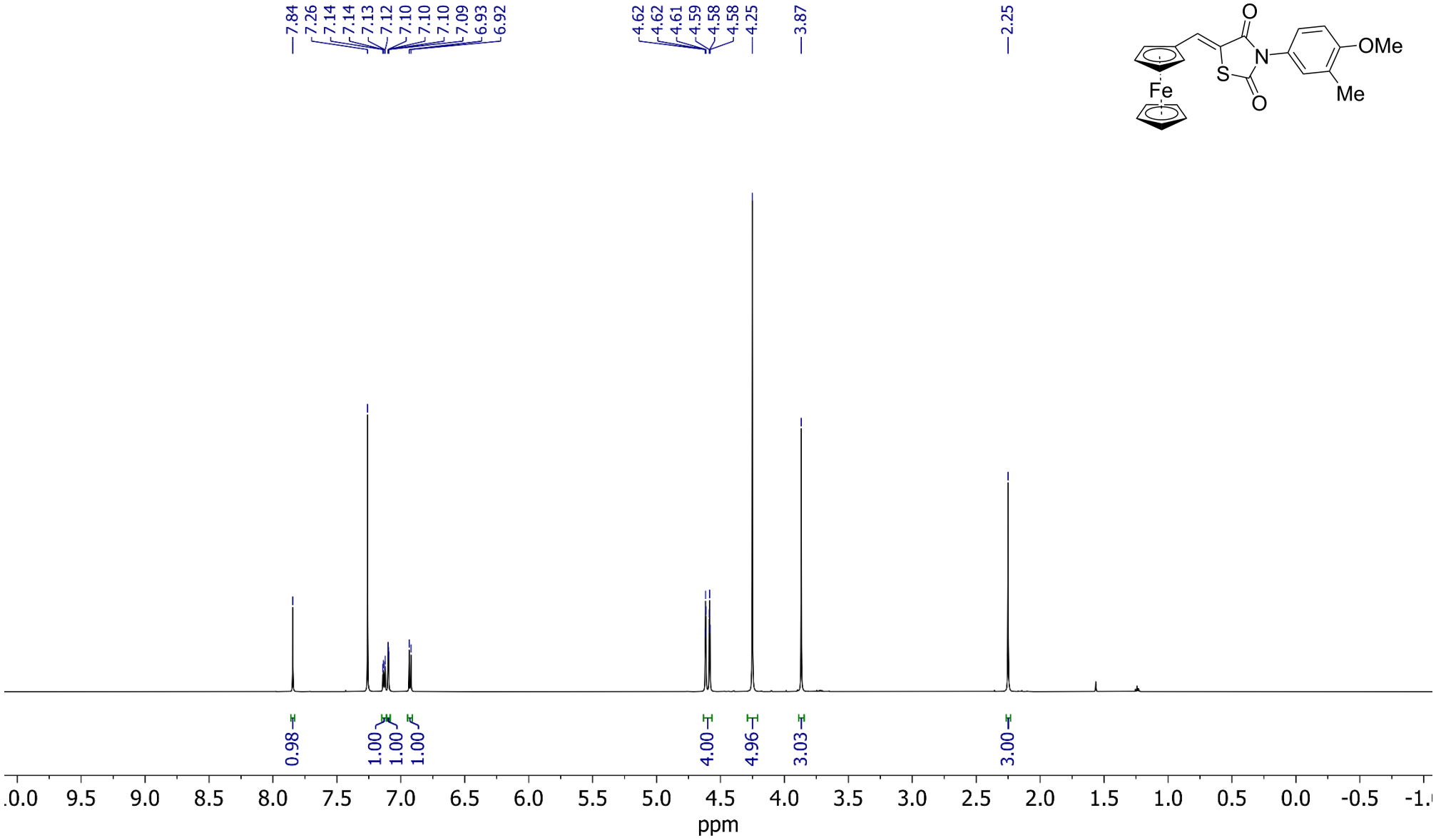
^{1}H NMR spectrum of 7b (600 MHz, $CDCl_3$)



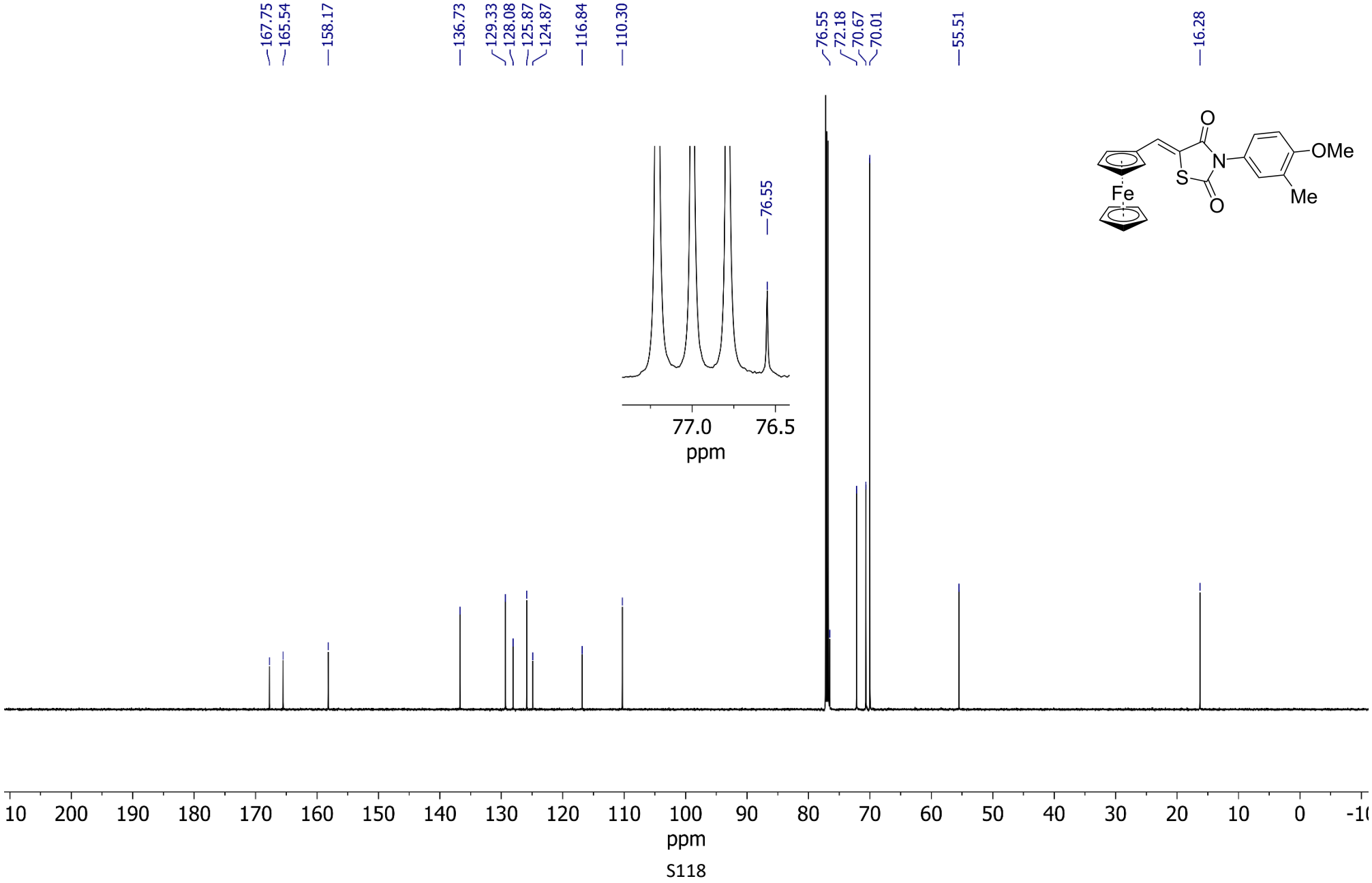
^{13}C NMR spectrum of 7b (151 MHz, $CDCl_3$)



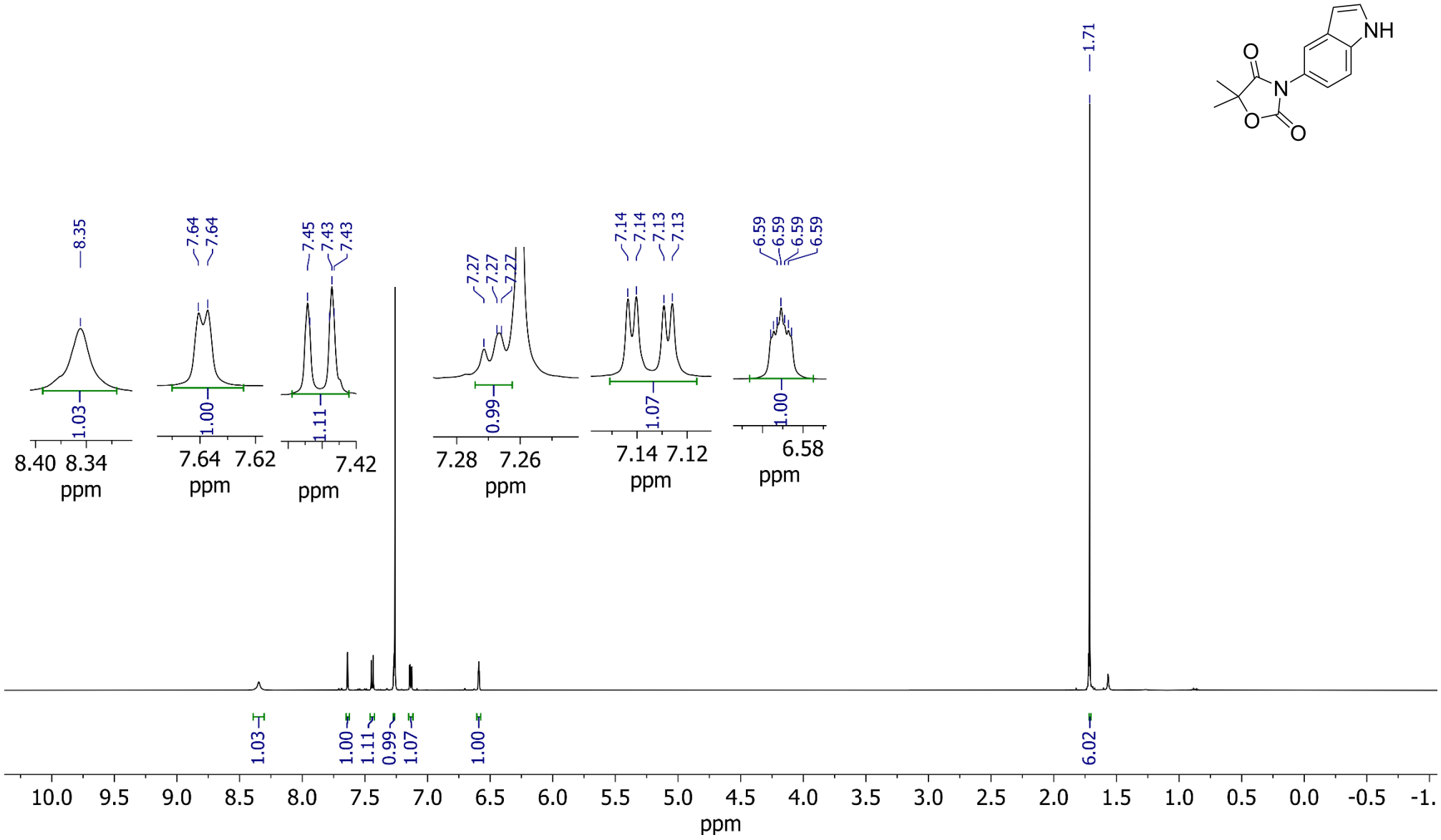
^{1}H NMR spectrum of 7e (600 MHz, $CDCl_3$)



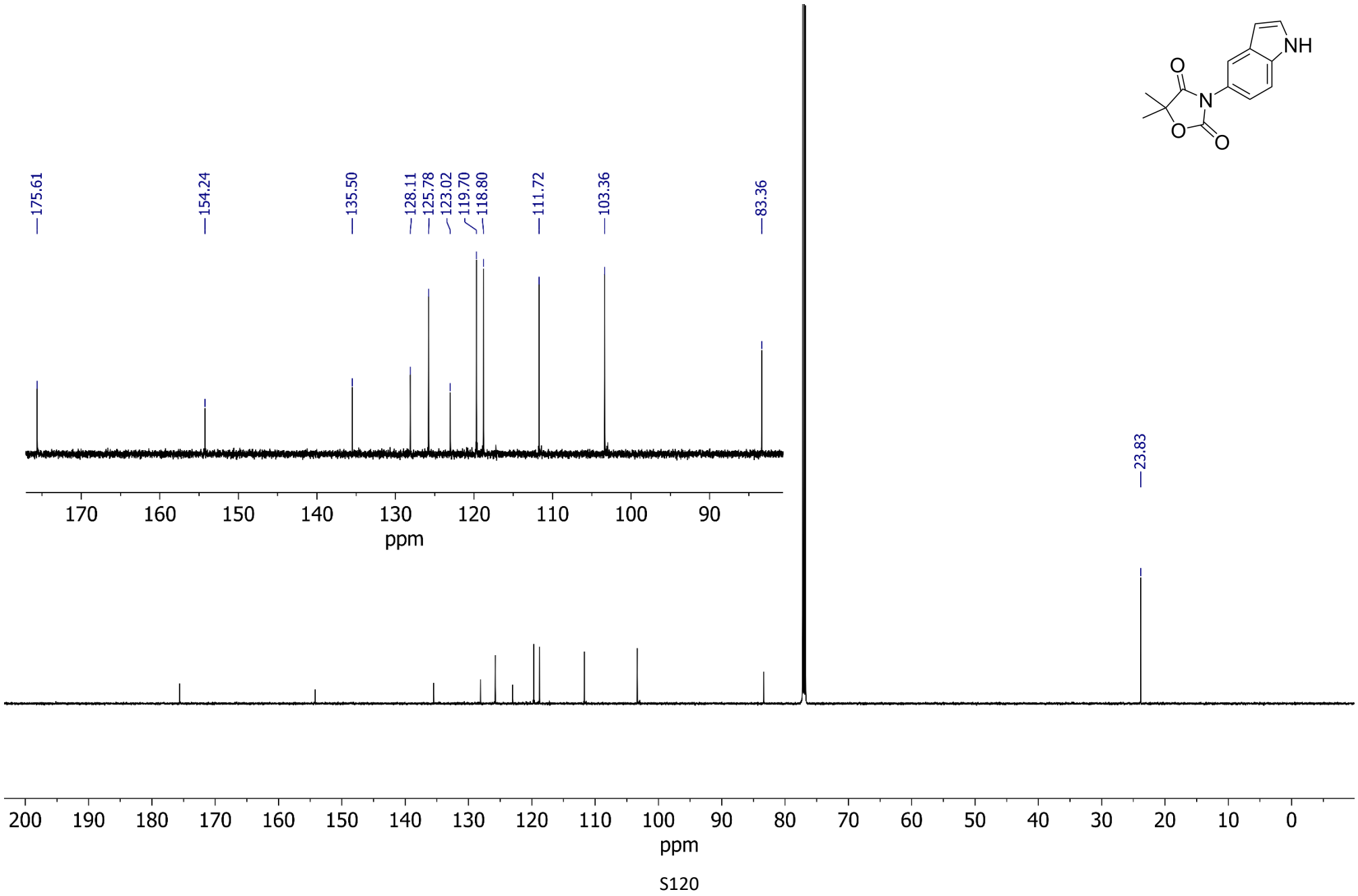
^{13}C NMR spectrum of 7e (151 MHz, $CDCl_3$)



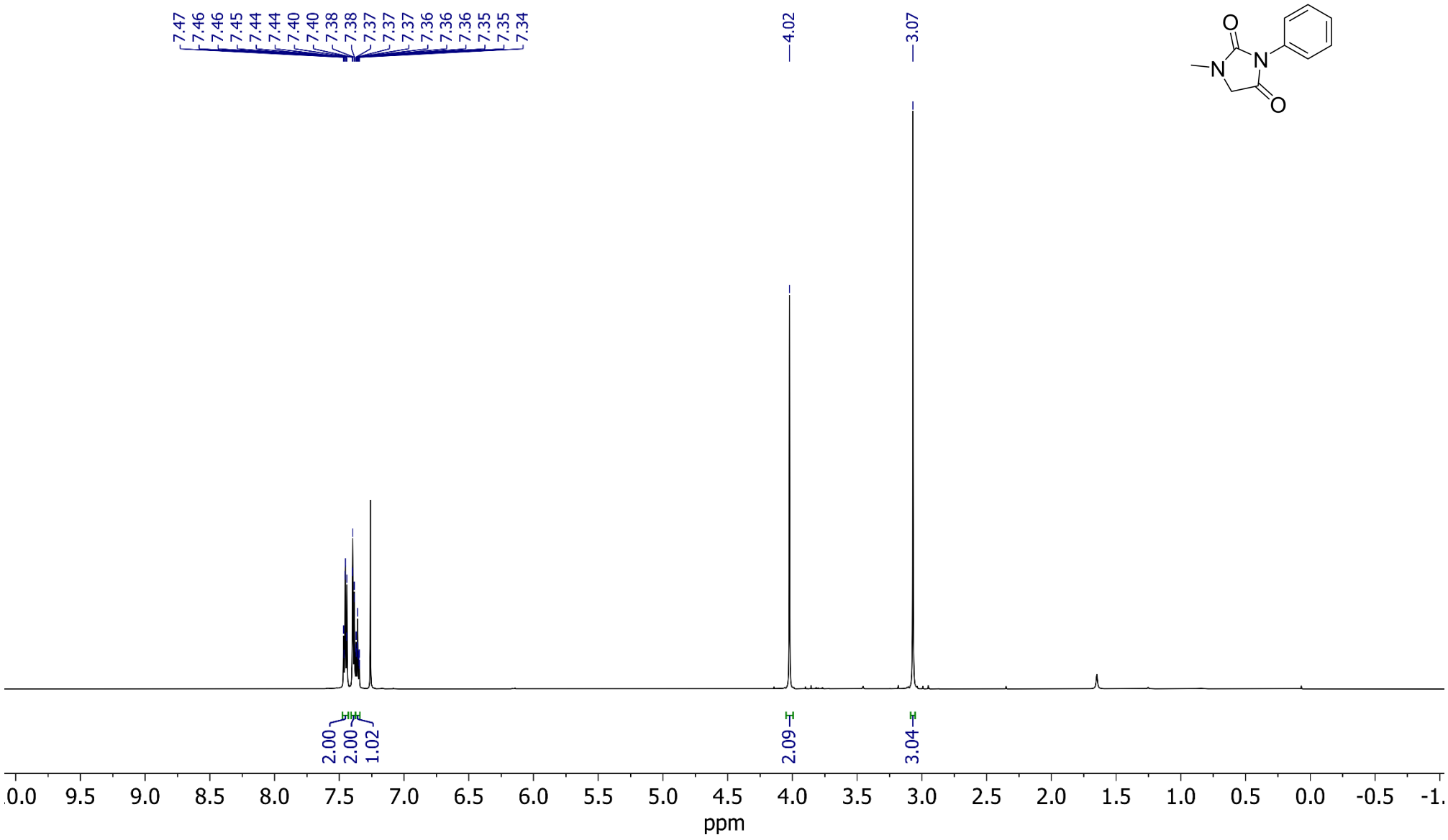
^{1}H NMR spectrum of 7f (600 MHz, $CDCl_3$)



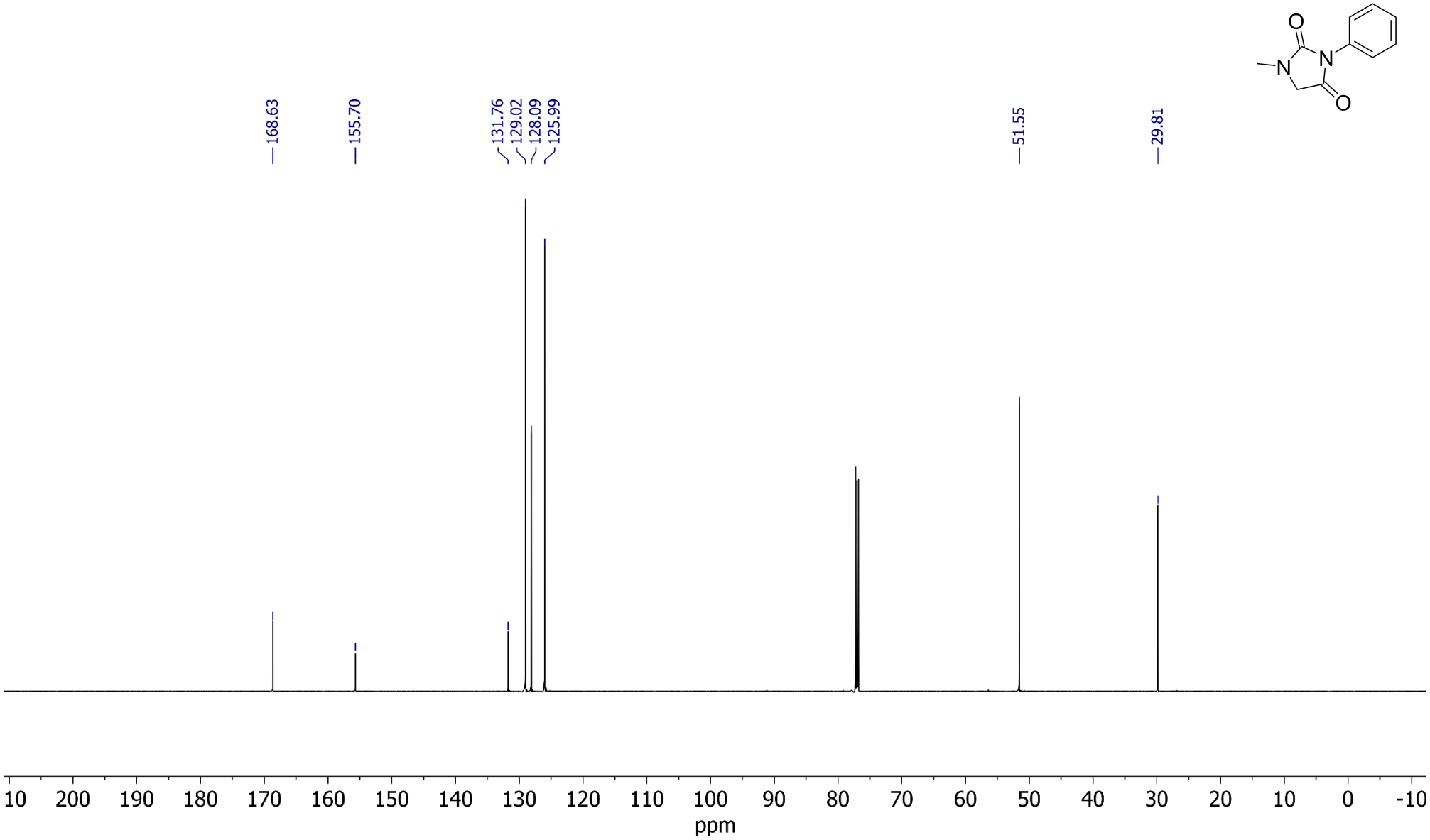
^{13}C NMR spectrum of 7f (151 MHz, $CDCl_3$)



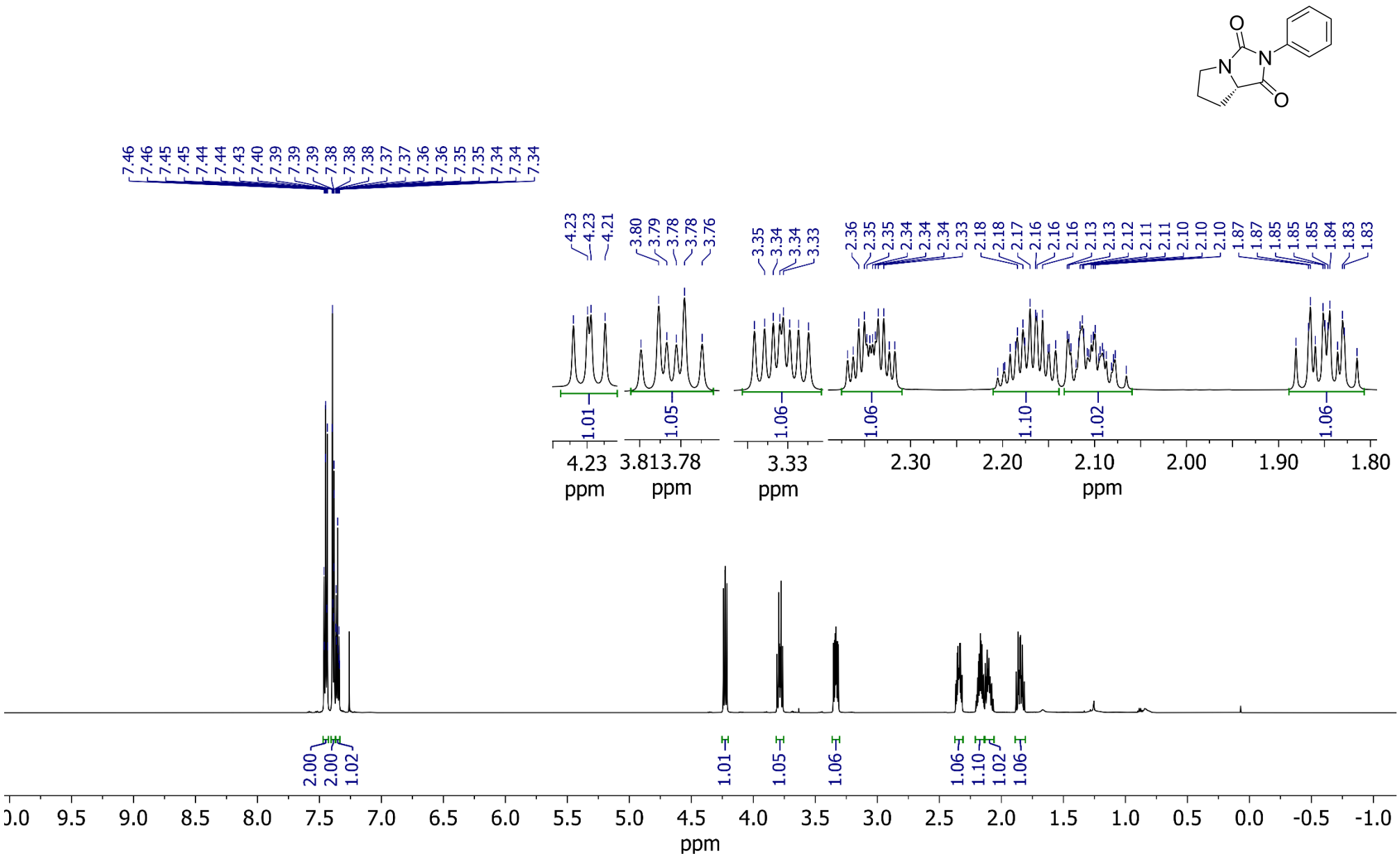
^{1}H NMR spectrum of 7g (600 MHz, $CDCl_3$)



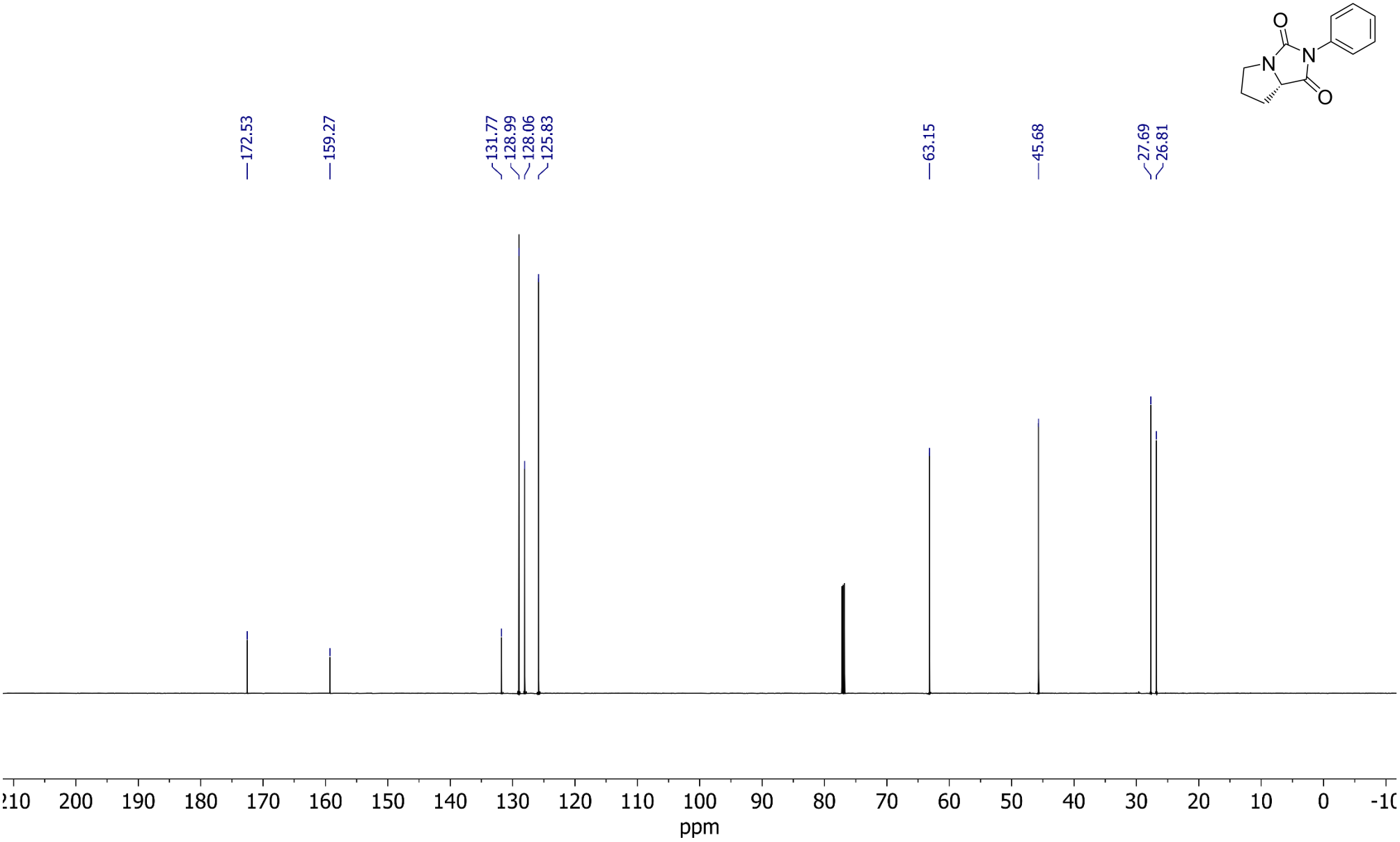
^{13}C NMR spectrum of 7g (151 MHz, $CDCl_3$)



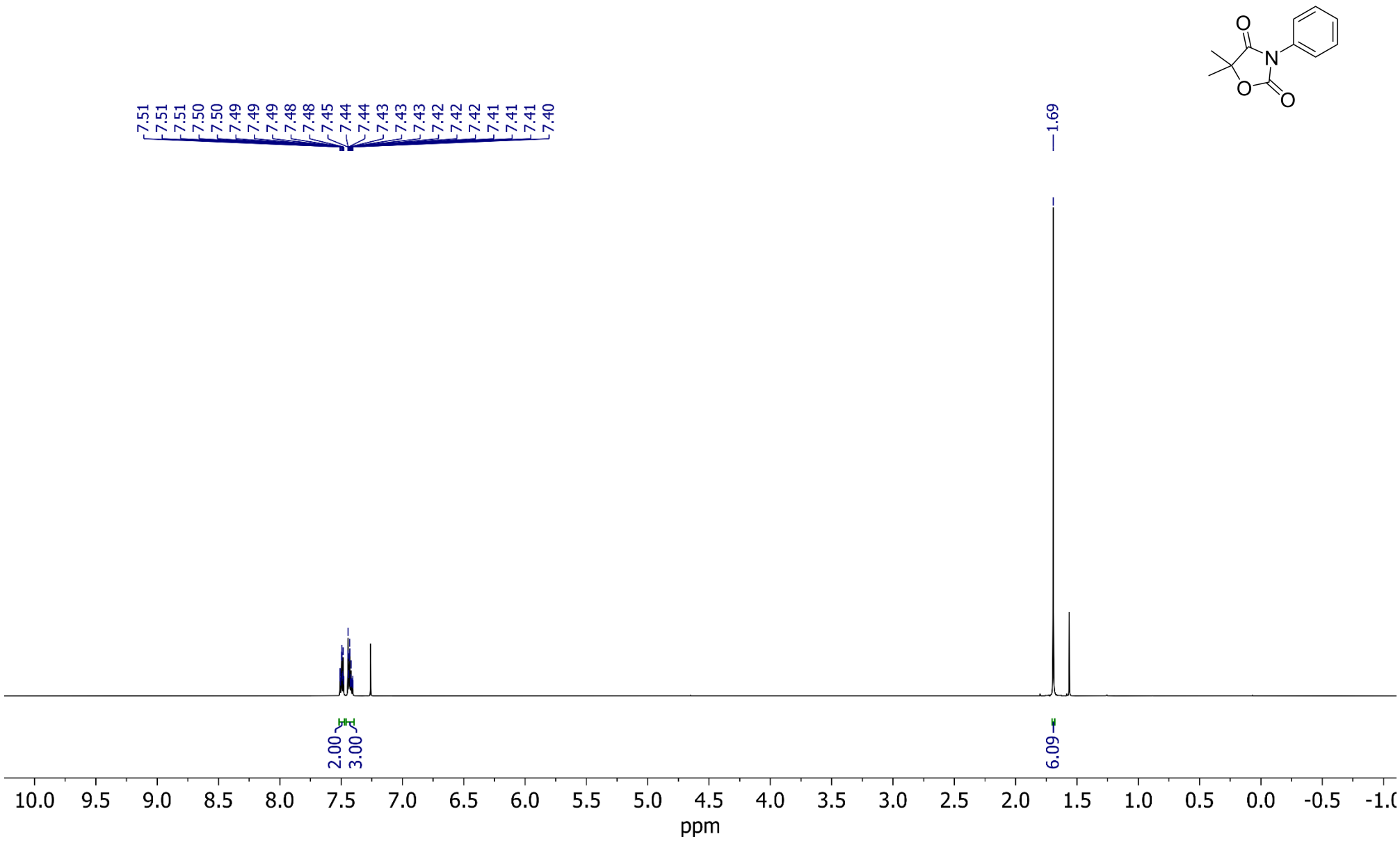
^{1}H NMR spectrum of 7h (600 MHz, $CDCl_3$)



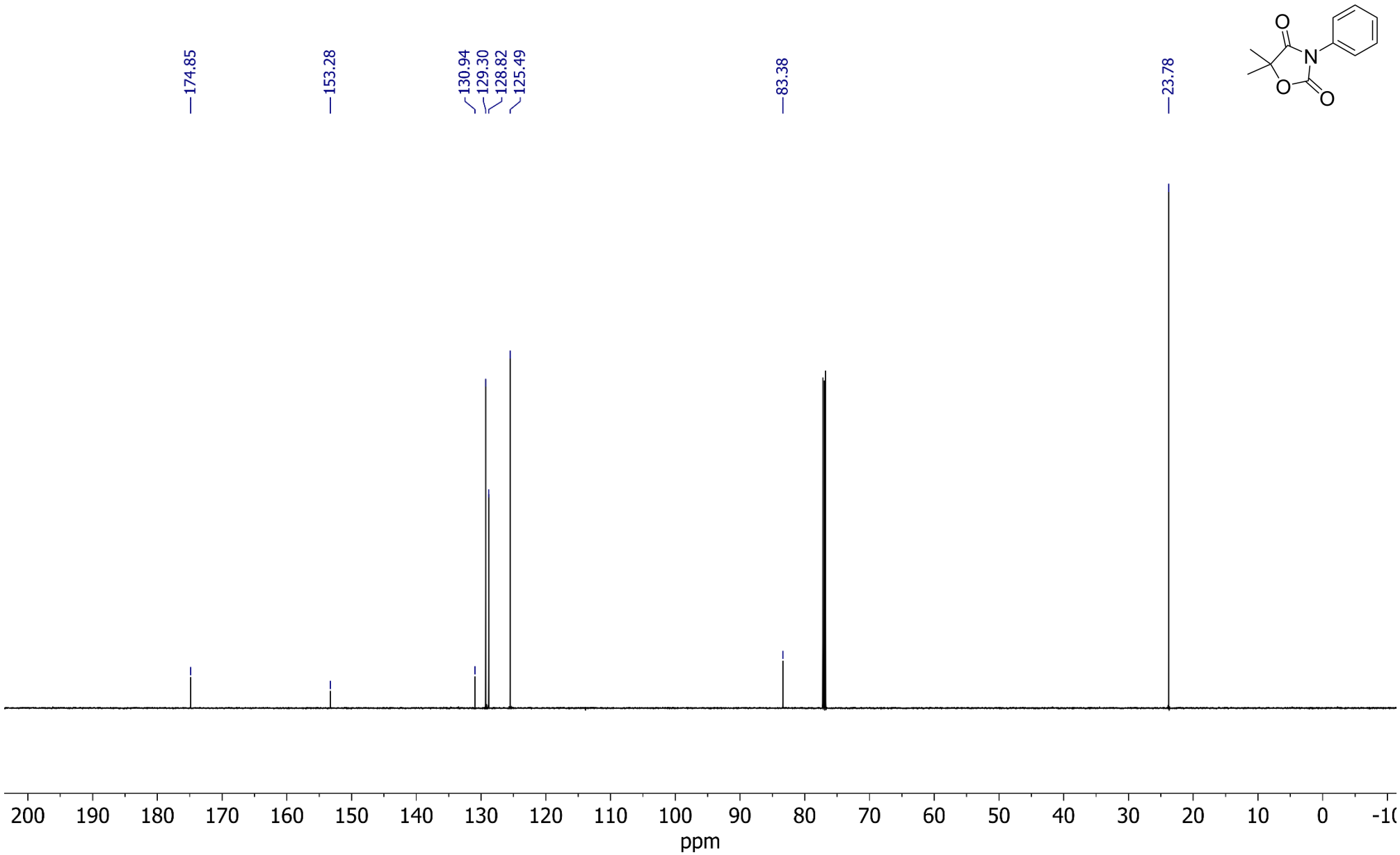
^{13}C NMR spectrum of 7h (151 MHz, $CDCl_3$)



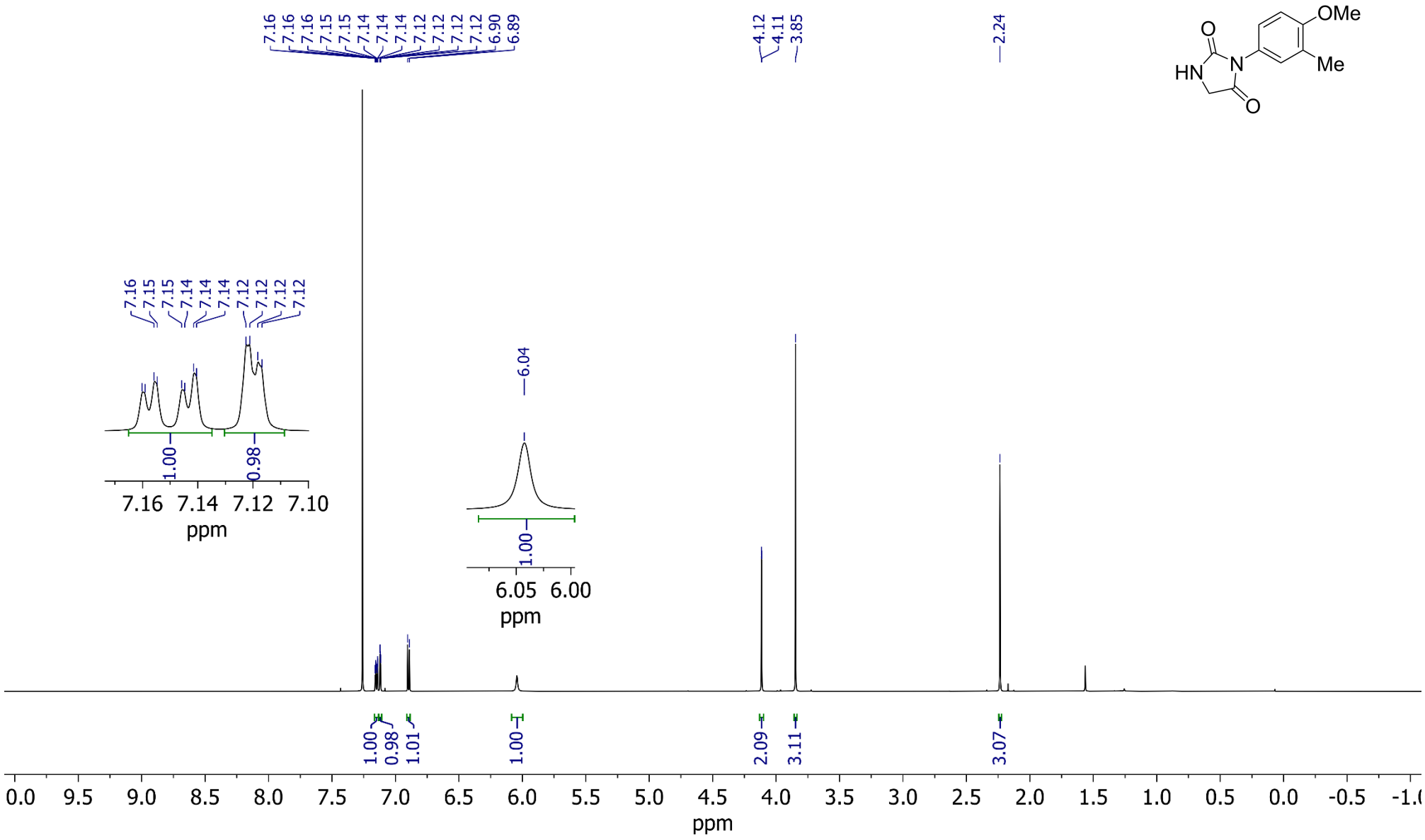
^{1}H NMR spectrum of 7i (600 MHz, $CDCl_3$)



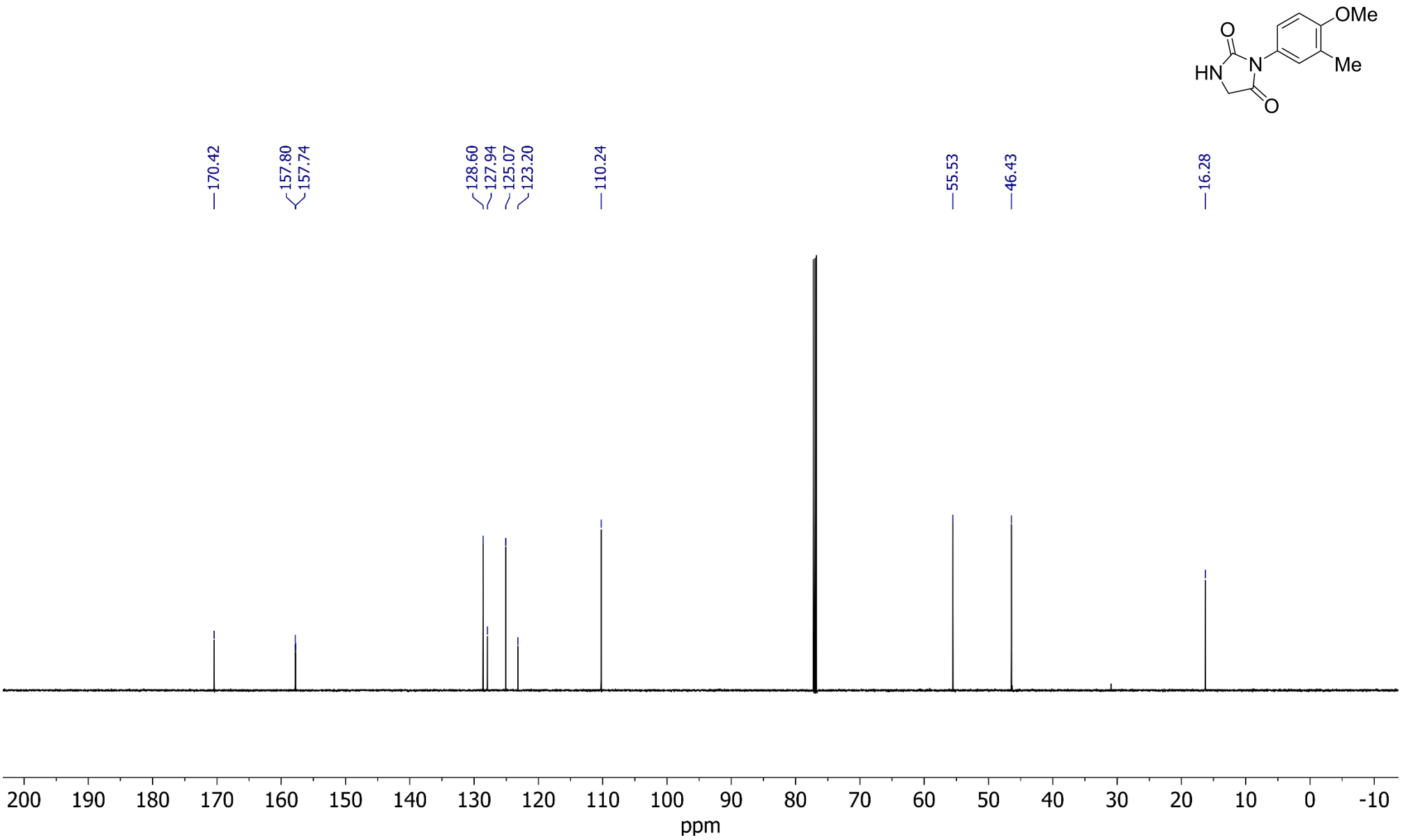
^{13}C NMR spectrum of 7i (151 MHz, $CDCl_3$)



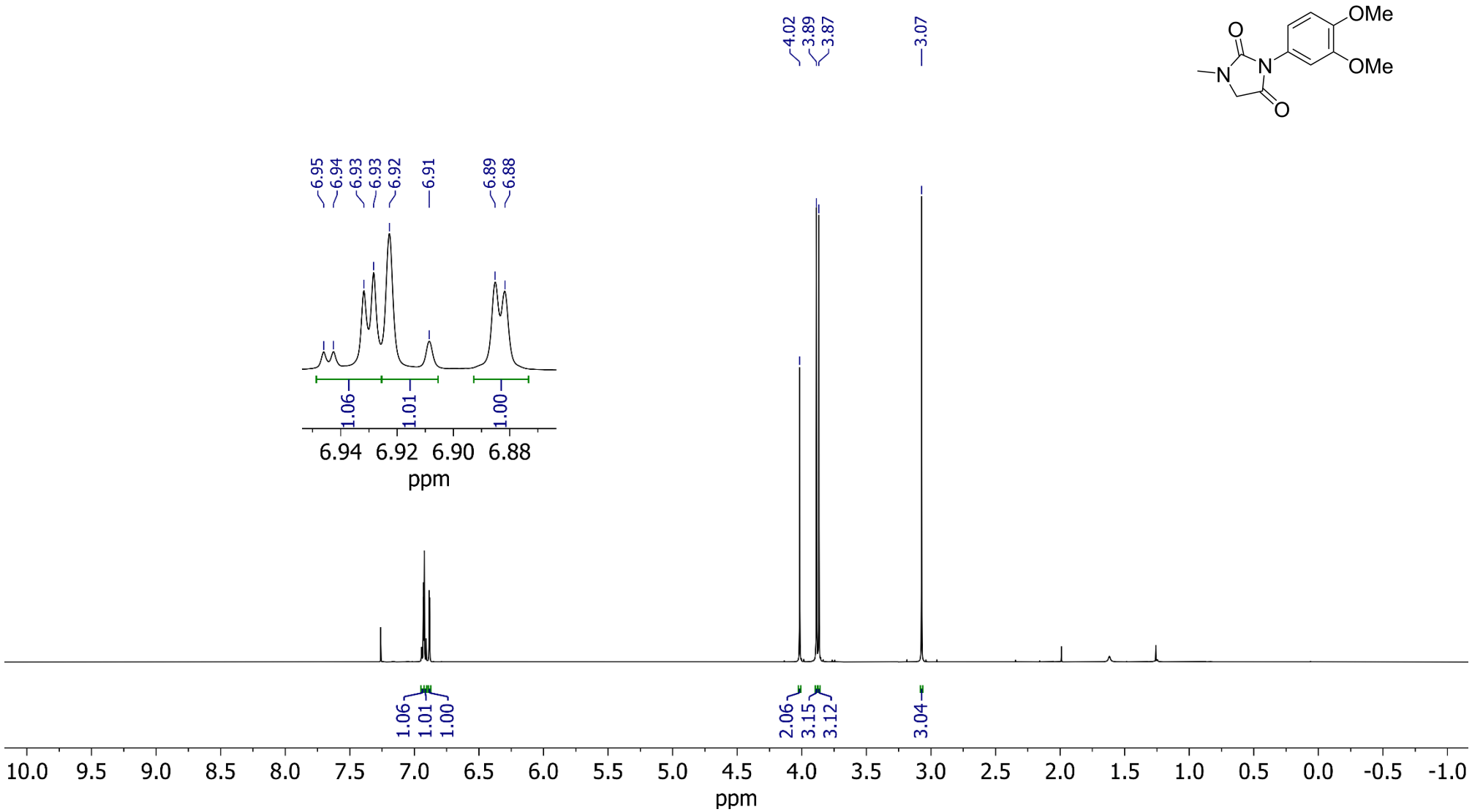
^{1}H NMR spectrum of 7j (600 MHz, $CDCl_3$)



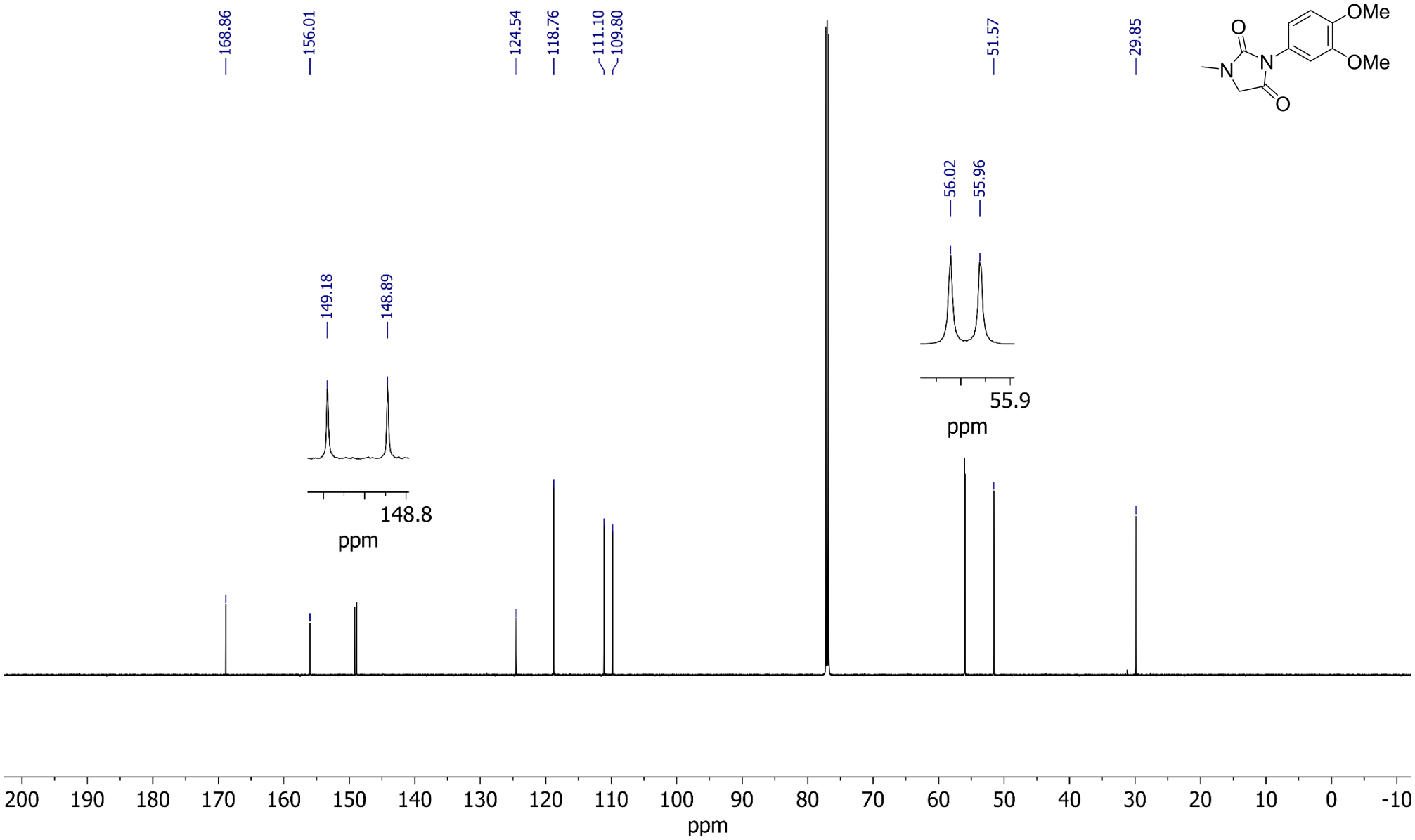
^{13}C NMR spectrum of 7j (151 MHz, $CDCl_3$)



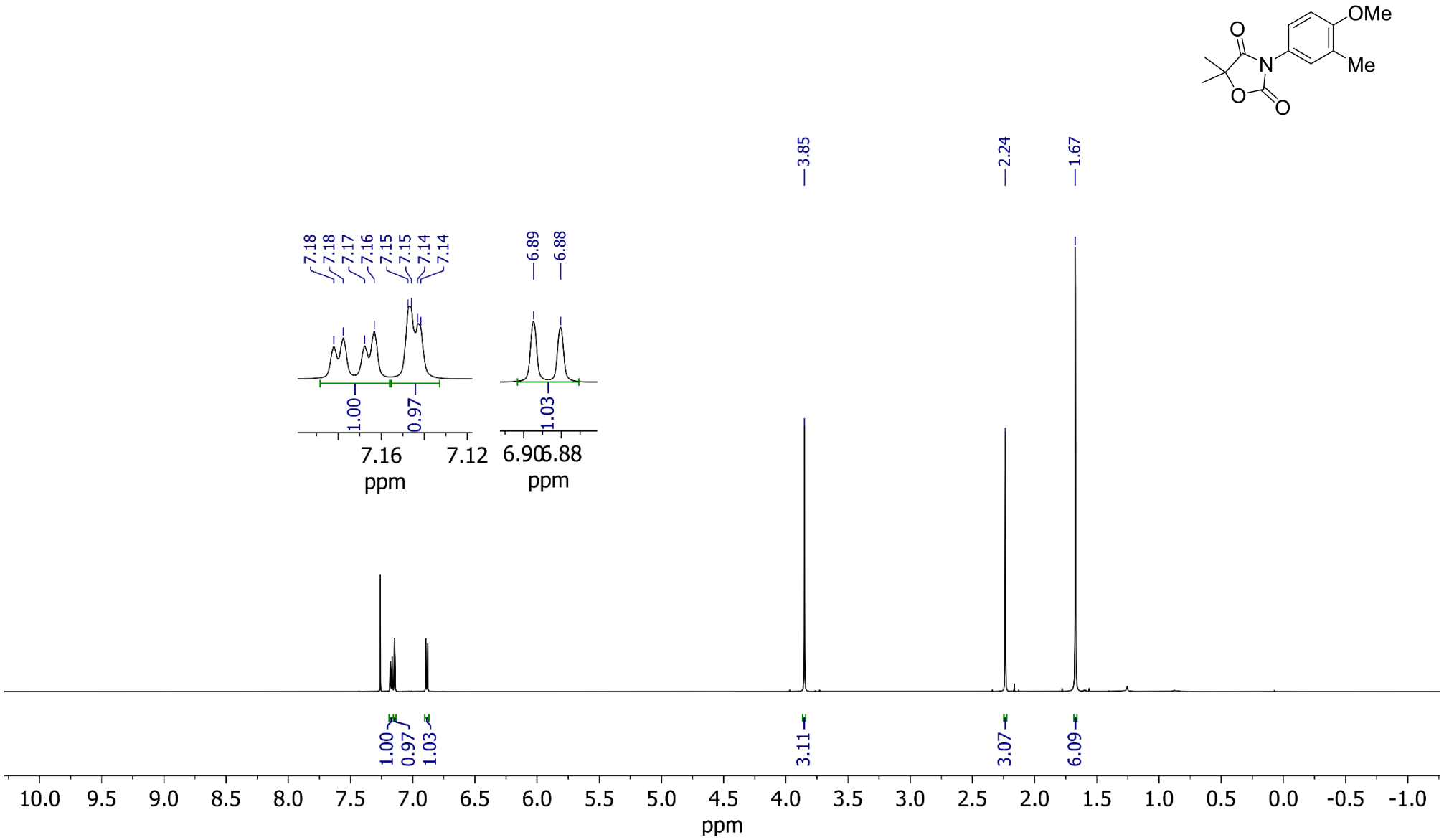
^{1}H NMR spectrum of 7k (600 MHz, $CDCl_3$)



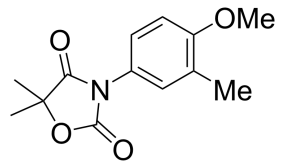
^{13}C NMR spectrum of 7k (151 MHz, $CDCl_3$)

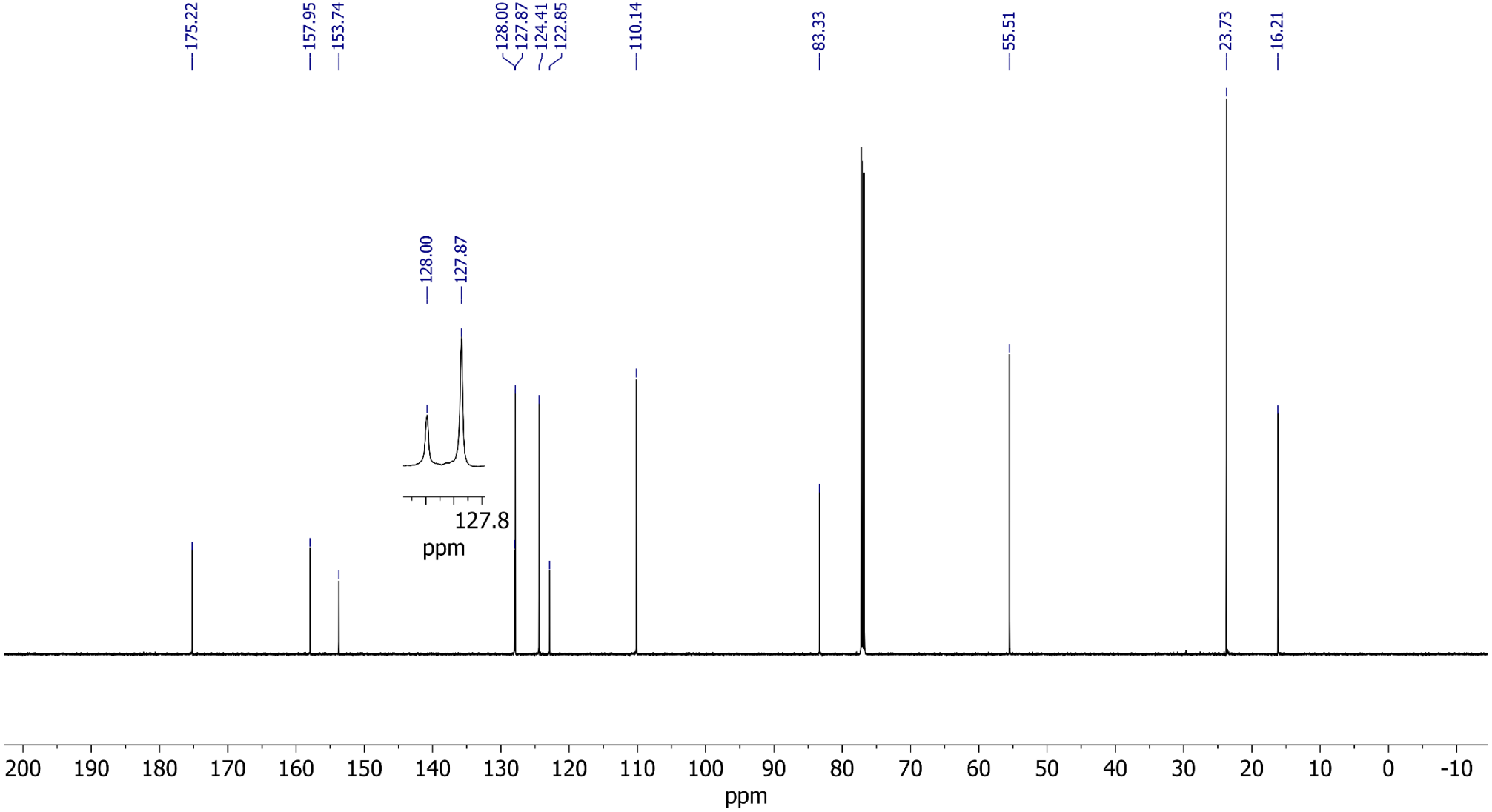


^{1}H NMR spectrum of 7l (600 MHz, $CDCl_3$)

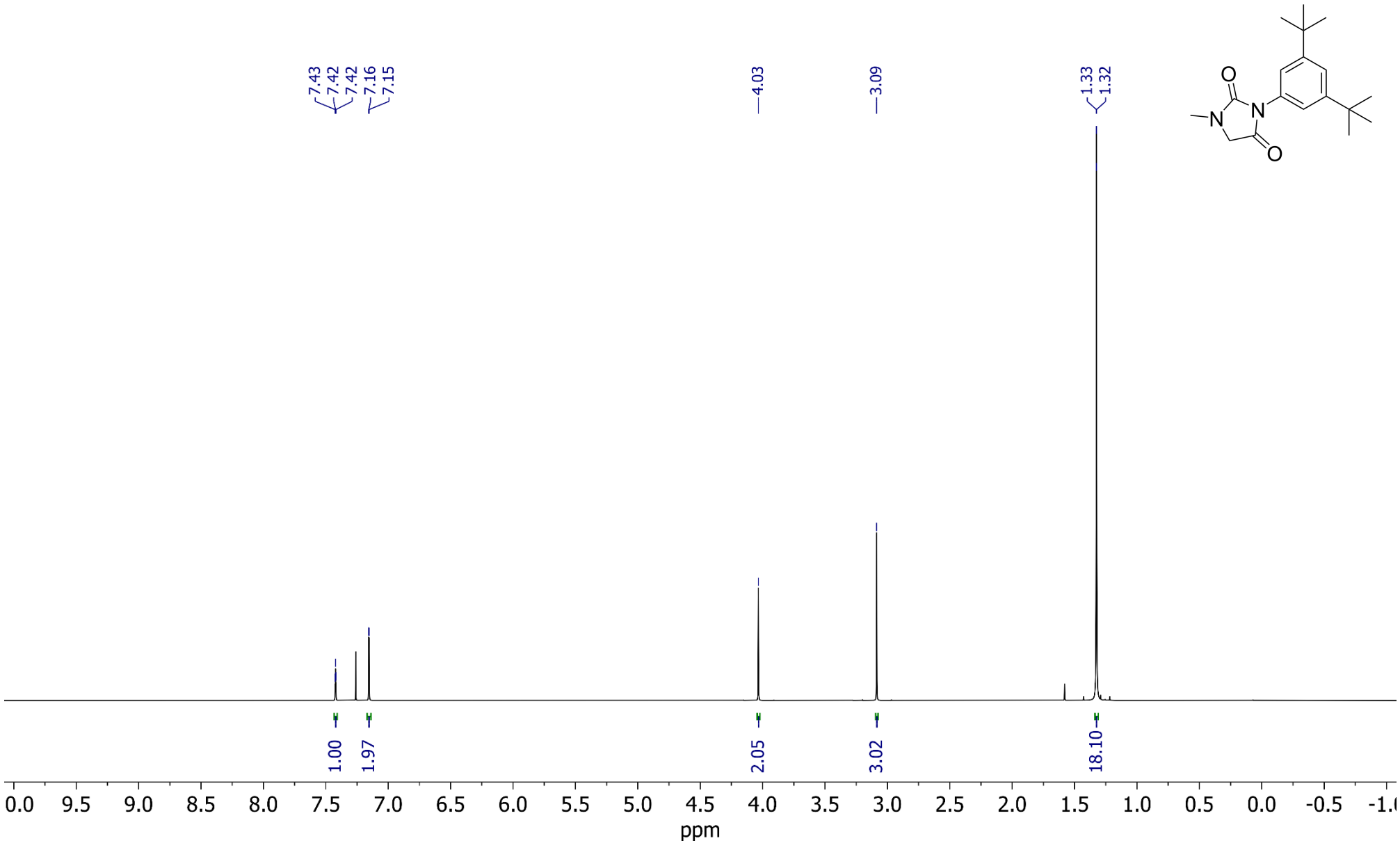


^{13}C NMR spectrum of 7l (151 MHz, $CDCl_3$)

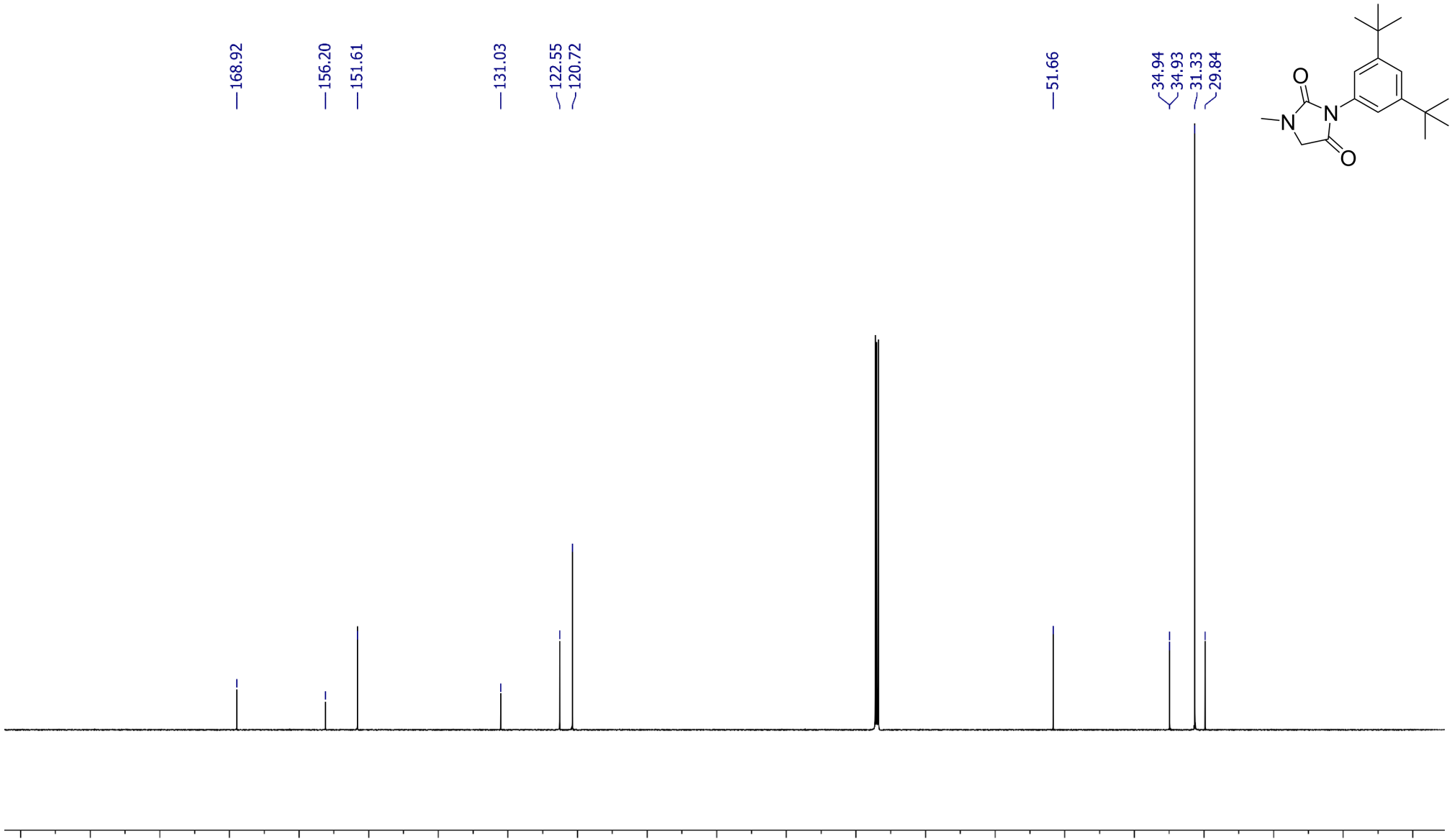




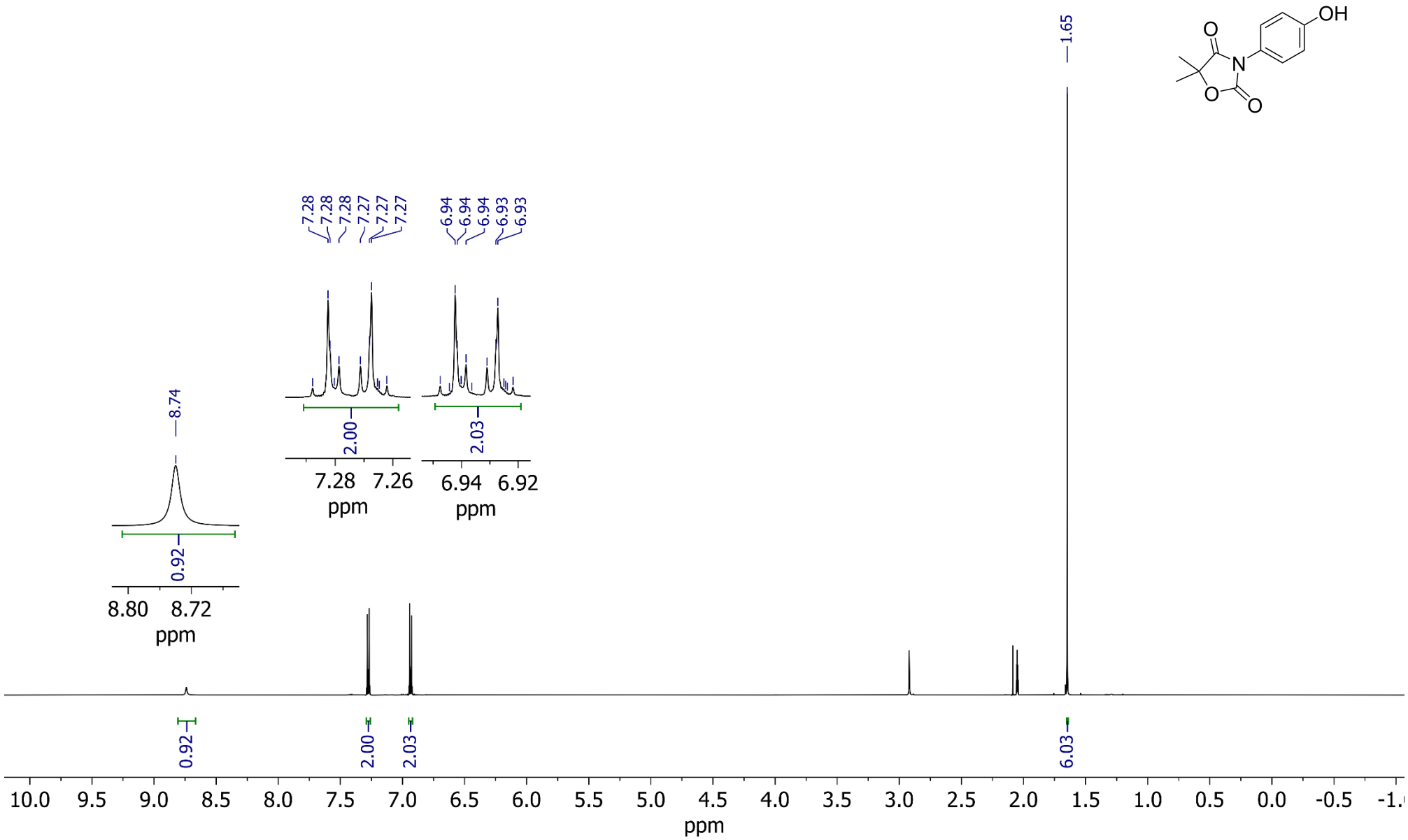
^{1}H NMR spectrum of 7m (600 MHz, $CDCl_3$)



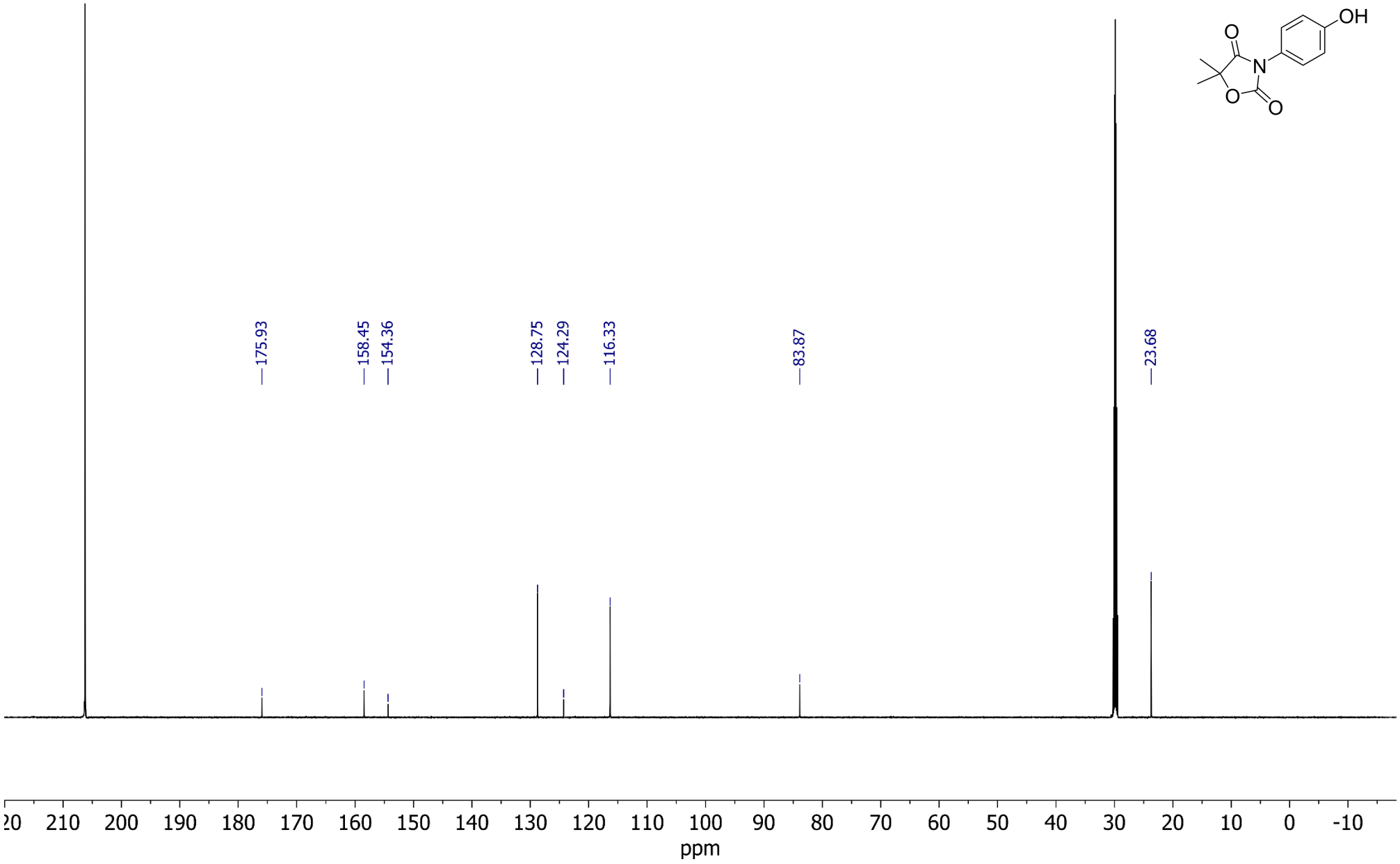
^{13}C NMR spectrum of 7m (151 MHz, $CDCl_3$)



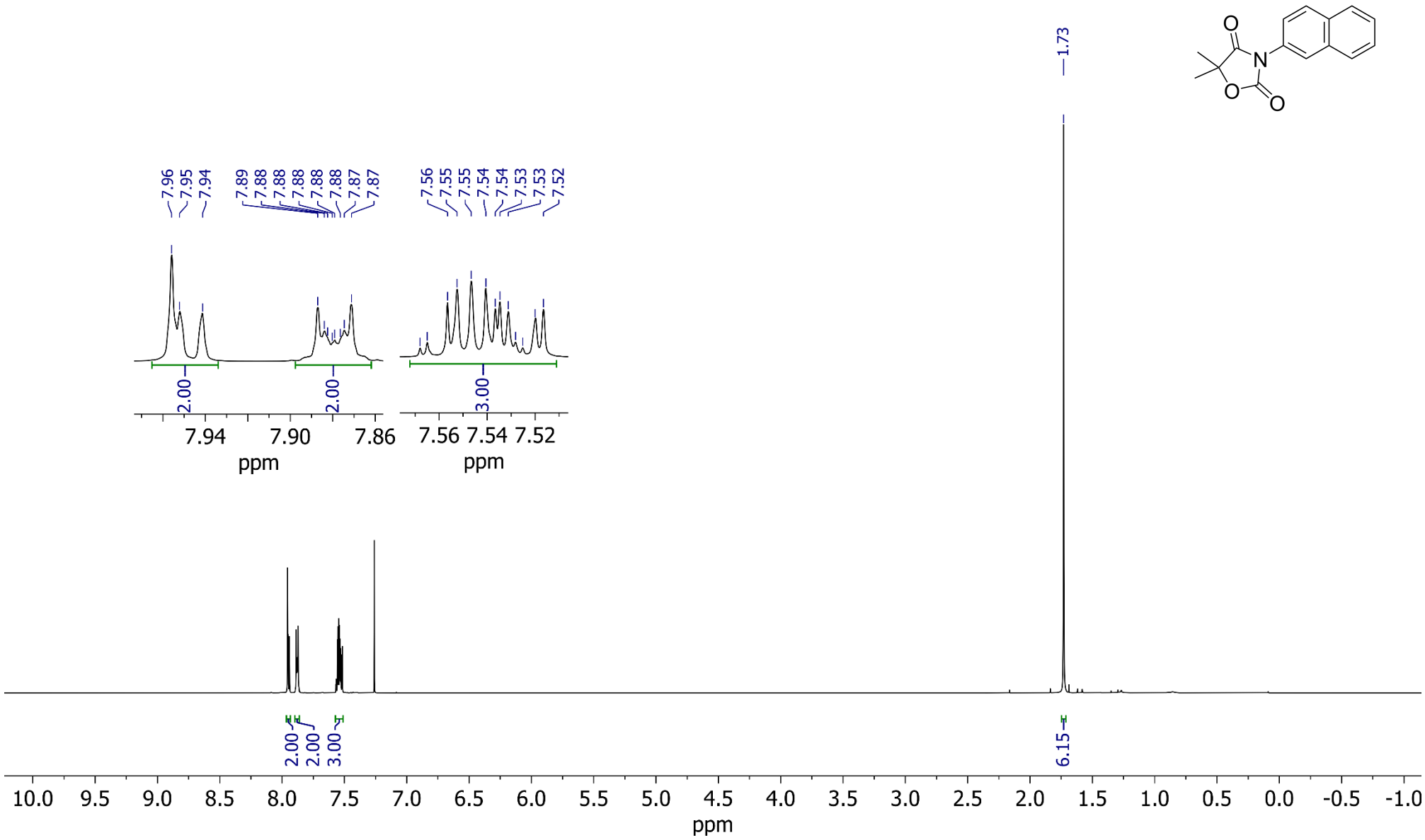
^{1}H NMR spectrum of 7n (600 MHz, Acetone-d_6)



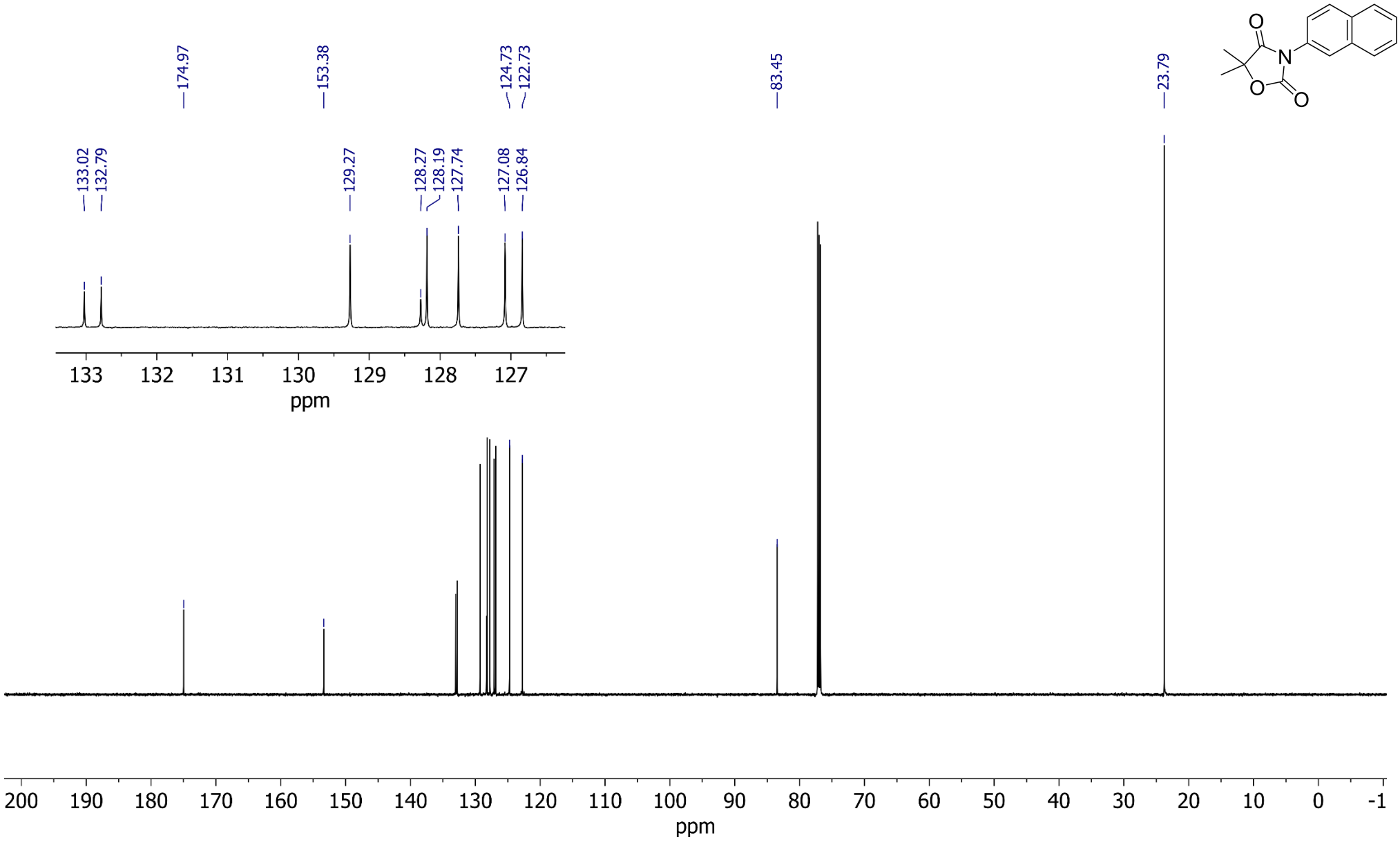
^{13}C NMR spectrum of 7n (151 MHz, Acetone-d_6)



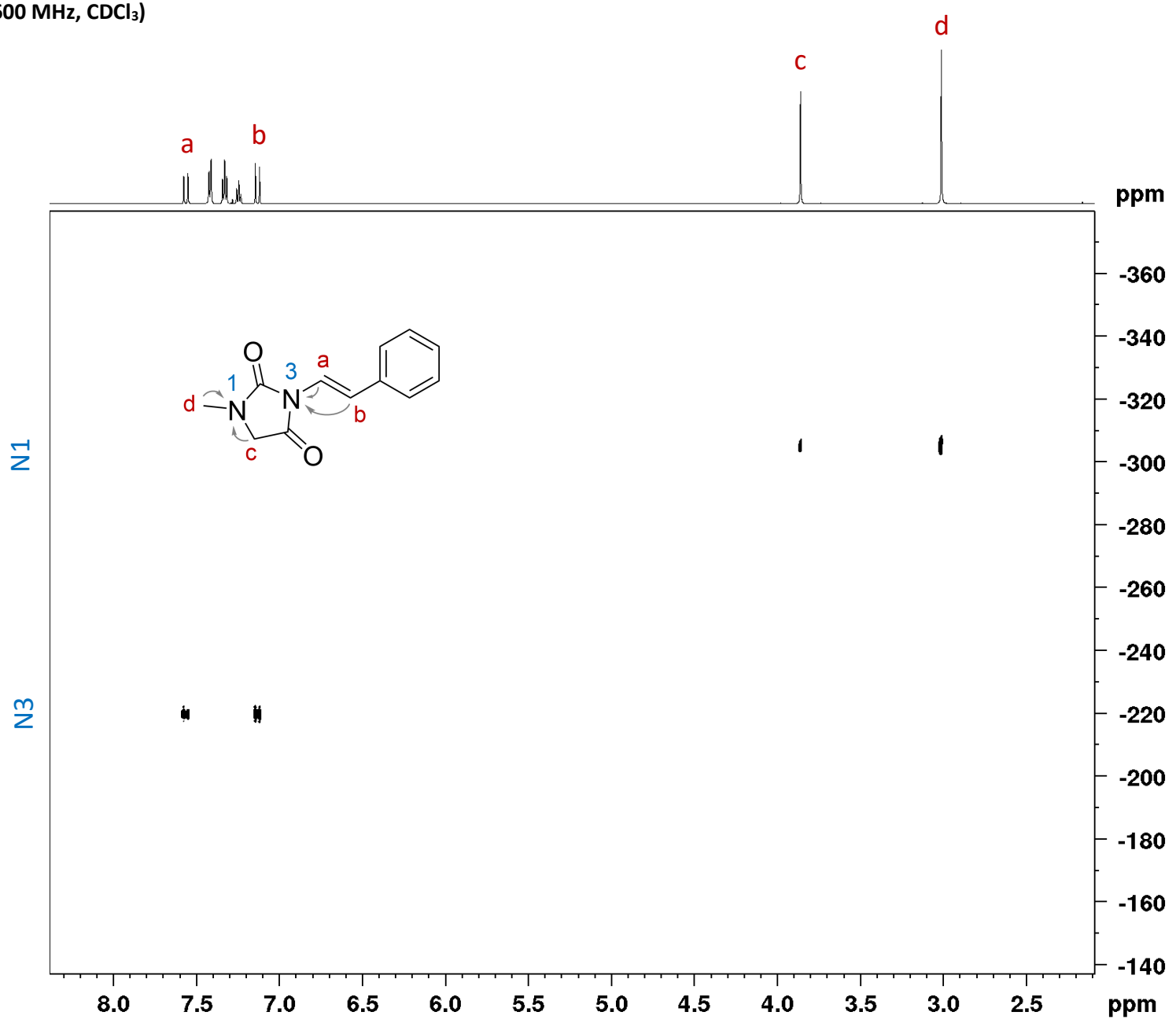
^{1}H NMR spectrum of 7o (600 MHz, $CDCl_3$)



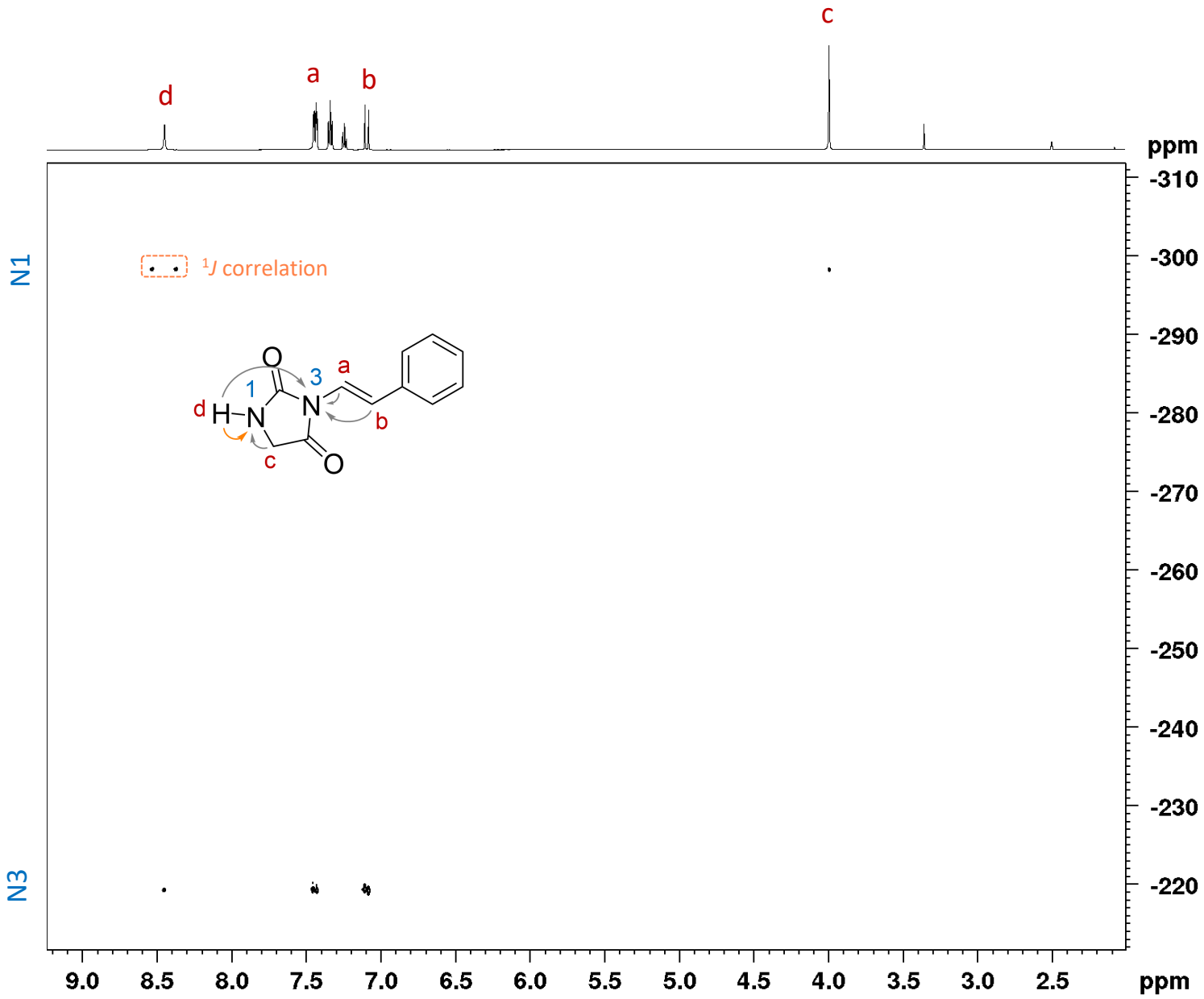
^{13}C NMR spectrum of 7o (151 MHz, $CDCl_3$)



^{1}H-^{15}N HMBC of 3a (600 MHz, $CDCl_3$)



^{1}H-^{15}N HMBC of 3e (600 MHz, $CDCl_3$)



^{1}H-^{15}N HMBC of 3s (600 MHz, CD_3CN)