

**Nikel -promoted oxidative domino C_{sp3}-H/N-H bond
double-isocyanide insertion reaction to construct pyrrolin-2-ones**

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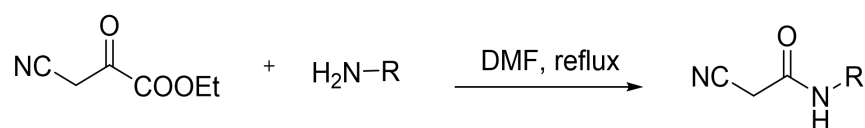
Experimental Section

1.1 General Methods

All reagents and solvents were obtained from commercial suppliers and used without further purification unless otherwise specified. 1,4-dioxane was distilled from Na prior to use. All reagents were weighed and handled in air at room temperature. Melting points were recorded on a microscopic melting apparatus and uncorrected. ^1H NMR and ^{13}C NMR spectra were recorded at 500 MHz, and 125 MHz in CDCl_3 . Chemical shifts are reported in δ (ppm) relative to TMS. IR spectra were recorded on a FT-IR spectrometer and only major peaks are reported in cm^{-1} . HRMS spectra were performed on two different spectrometers with an ESI source. The X-ray single-crystal diffraction was performed on CCD area detector.

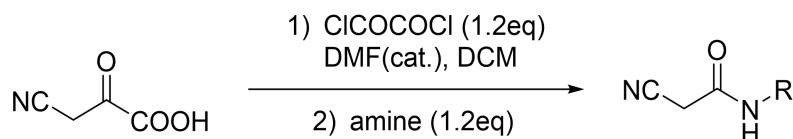
1.2 Preparation of Substrate 1

1.2.1 Preparation of Cyanoacetamides.



R = Ph, Bn, Cy, *p*- FC_6H_4 -, *p*- ClC_6H_4 -, *p*- BrC_6H_4 -, *p*- MeOC_6H_4 -, *p*- MeC_6H_4 -, *m*- ClC_6H_4 -, *o*- ClC_6H_4 -, Cyclopropyl, Naphthyl, 2-biphenyl, 1-piperonyl

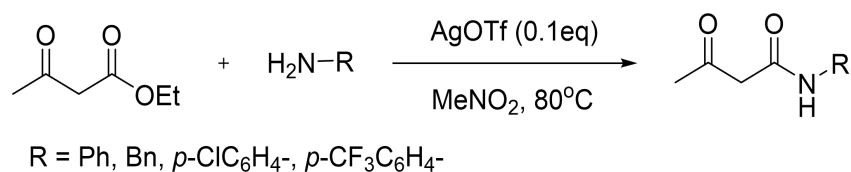
Method A^[1]. A mixture of amine (10 mmol) and ethyl cyanoacetate (11 mmol, 1.244 g) in DMF (20mL) was refluxed for 5 h and then left to cool to room temperature. The reaction mixture was then poured onto ice-cold water and the precipitated solid was filtered off and recrystallized from ethanol to afford cyanoacetamide.



amine = 2-amino-pyridin, 4-(trifluoromethyl)aniline

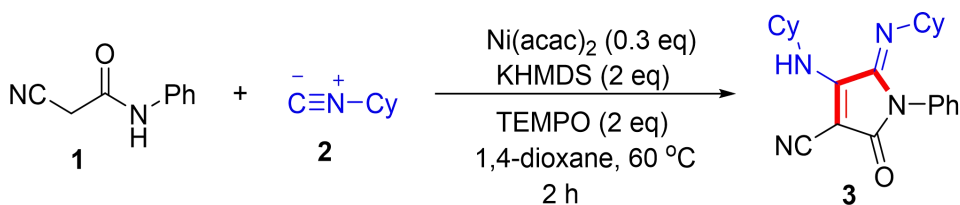
Method B^[2]. To a dried flask were added acid (10.0mmol, 0.851 g), DCM (20mL), and three drops of DMF sequentially. Then oxalyl chloride (1.2mL, 12.0mmol) was added dropwise within 5 min at 0 °C. The resulting mixture was stirred at room temperature for 3.5 h and then added amine (12 mmol) and water (20 mL). The resulting mixture was stirred for 3h at rt, the completion of the reaction was monitored by TLC. Removal of the solvent in vacuo then ice-cold water was added. Following by a simple filtration and washing procedure with petroleum ether to result in cyanoacetamide.

1.2.2 Preparation of β -Oxo Amides^[3].



An oven-dried 250 mL round-bottomed flask was charged with ethyl acetoacetate (10 mmol), amine (10 mmol), silver triflate (1.0 mmol, 0.256 g) and nitromethane (50 mL). After stirring at 80 °C for 8 h, the completion of the reaction was monitored by TLC. Removal of the solvent in vacuo and purification of the residue by silica gel column chromatography afforded the desired product.

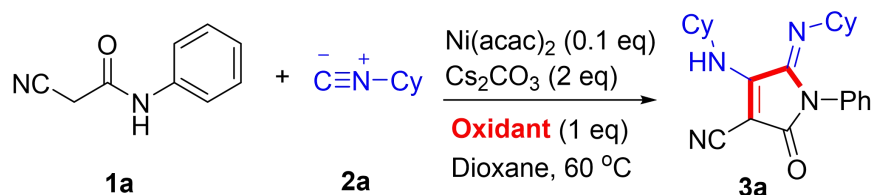
1.3 General Procedure For Pyrrolin-2-Ones (**3**)



A dried 10 mL round-bottomed flask was charged with cyanoacetamide **1** (42,0 mg, 0.50 mmol), Ni(acac)₂ (38.5 mg, 0.15 mmol), TEMPO (156.3 mg, 1.0 mmol), isocyanocyclohexane **2** (136.5 mg, 1.25 mmol), 1,4-dioxane (1 mL) and KHMDS (1.0 mol/L in THF, 1 mL, 1.0 mmol). The reaction mixture was stirred at 60 °C in air for 2h. After completion of the reaction as monitored by TLC. Removal of the solvent in vacuo and purification of the residue by silica gel column chromatography(petroleum ether/EtOAc, 15:1 then 4:1, v/v), then washing with petroleum ether afforded the desired product.

1.4 Optimization of Reaction Conditions

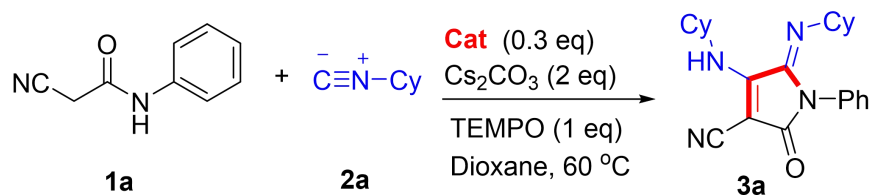
Oxidant^a



Entry	Oxidant	Time (h)	% yield 3aa
1	air	12	46
2	O ₂	7	38
3	CuCl ₂	12	trace
4	DCP	12	41
5	DTPB	5	46
6	oxone	12	31
7	H ₂ O ₂	12	42
8	TBHP	12	40
9	TEMPO	4	51

^aConditions: 0.6 mmol of **1a**, 1.5 mmol of **2a**, 10 mol% of Ni(acac)₂, 1.2 mmol of Cs₂CO₃, Dioxane (1.2 mL), air, 60°C.

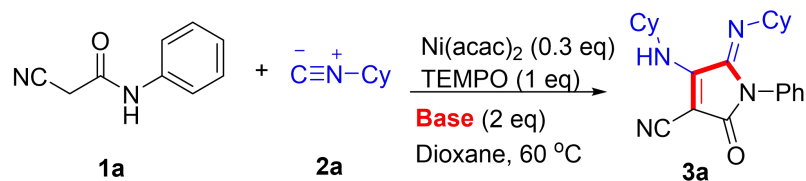
Metal catalyst^a



Entry	Cat	Time (h)	Yield (%)
1	—	12	N.R.
2	(Cp* <i>Rh</i> Cl ₂) ₂	12	N.P.
3	Pd(OAc) ₂	3.5	19
4	Pd(PPh) ₂ Cl ₂	3.5	21
5	Co(acac) ₂	3.5	7
6	Co(acac) ₃	3.5	20
7	Ni(acac)₂	3.5	34
8	Ni(PPh) ₂ Cl ₂	3.5	9
9	NiCl ₂ 6H ₂ O	3.5	21
10	Ni(OAc) ₂ 4H ₂ O	3.5	15
11	Ni(COD) ₂	12	trace

^aConditions: 0.6 mmol of **1a**, 1.5 mmol of **2a**, 1.2 mmol of Cs₂CO₃, 0.6 mmol of TEMPO, air, Dioxane (1.2 mL), air, 60°C. N. P = no product.

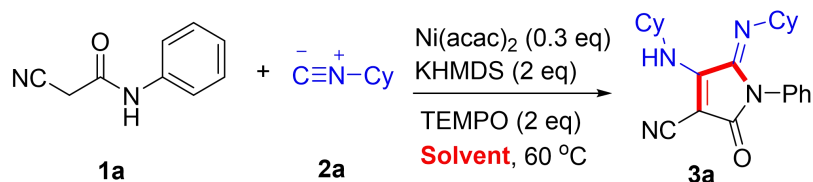
Base^a



Entry	Base	Time (h)	Yield(%)
1	Cs ₂ CO ₃	2.5	44
2	NaOH	3.5	58
3	LiOH	3.5	62
4	NaH	5	65
5	CaH	12	trace
6	<i>t</i> -BuOLi	3	49
7	<i>t</i> -BuONa	6	43
8	<i>t</i> -BuOK	6	37
9	KHMDS	2	84

^aConditions: 0.6 mmol of **1a**, 1.5 mmol of **2a**, 30 mol% of Ni(acac)₂, 0.6 mmol of TEMPO, air, Dioxane (1.2 mL), air, 60°C.

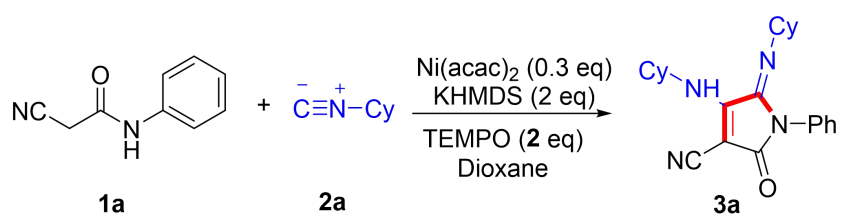
Solvent^a



Entry	Solvent	Time (h)	Yield(%)
1	toluene	4.5	39
2	fluorobenzene	8	61
3	chlorobenzene	7	70
4	DCE	11	21
5	CH ₃ CN	1.5	52
6	1,4-dioxane	2	84
7	THF	3	68
8	2-Me-THF	4	44

^aConditions: 0.6 mmol of **1a**, 1.5 mmol of **2a**, 30 mol% of Ni(acac)₂, 0.6 mmol of TEMPO, air, 1.2 mmol of KHMDS, air, 60°C.

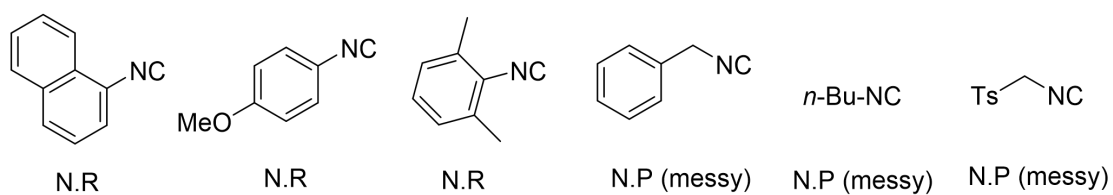
Temperature



Entry	Temp. (°C)	Time (h)	Yield (%)
1	25	24	47
2	40	8	70
3	60	2	84
4	80	1	60

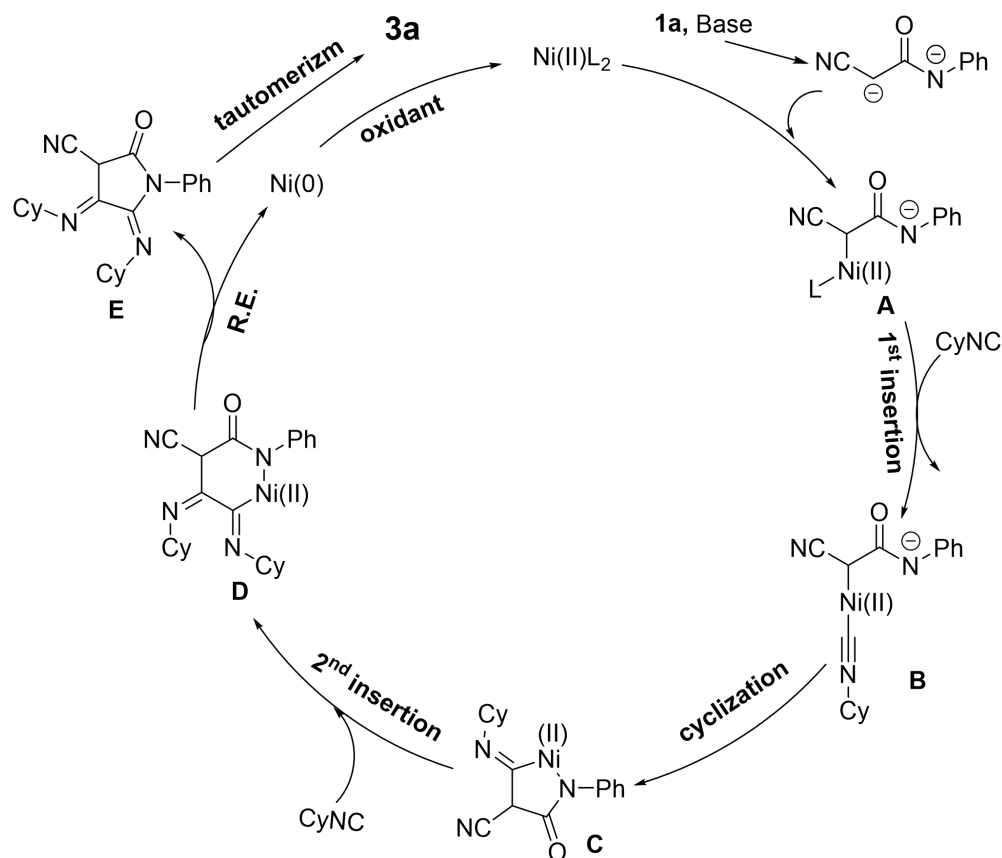
^aConditions: 0.6 mmol of **1a**, 1.5 mmol of **2a**, 30 mol% of $\text{Ni}(\text{acac})_2$, 0.6 mmol of TEMPO, air, 1.2 mmol of KHMDS, Dioxane (1.2 mL).

Ineffectual Isocyanides



N.R = no reaction; N.P = no product

1.5 Another Reaction Mechanism



2. References

- (1) Li, Z.; Zhu, A.; Yang, J. *J. Heterocycl. Chem.* 2012, 49, 1458.
- (2) Fisher, L. E.; Caroon, J. M.; Stabler, S. R.; Lundberg, S.; Muchowski, J. M. *J. Org. Chem.* 1993, 58, 3643.
- (3) Vandavasi, J. K.; Hsiao, C. T.; Hu, W. P.; Boominathan, S. S. K.; Wang, J. J. *European J. Org. Chem.* 2015, 2015 (14), 3171.

3. X-Ray Crystal Structure of 3d

(*Z*)-1-(4-bromophenyl)-4-(cyclohexylamino)-5-(cyclohexylimino)-2-oxo-2,5-dihydro-1*H*-pyrrole-3-carbonitrile (**3d**) (CCDC 1482888)

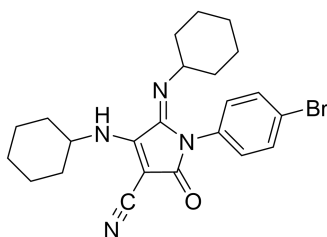
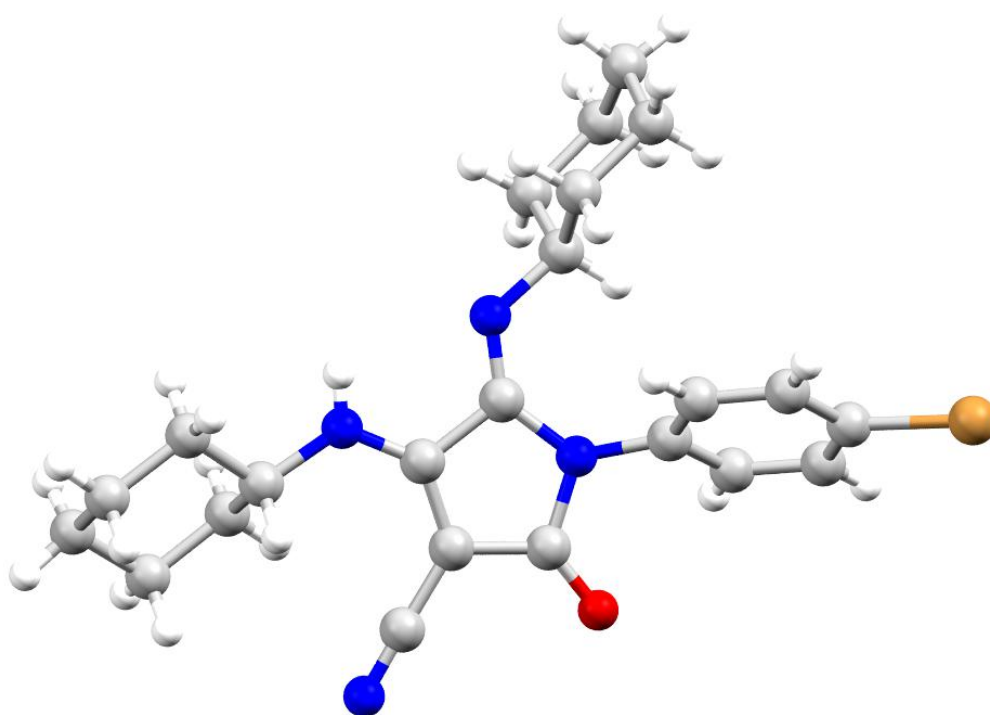


Figure S1. X-Ray crystal structure of **3d**, with 50% probability displacement ellipsoids

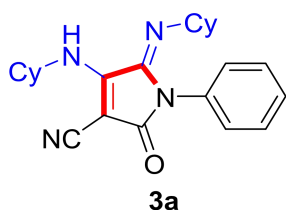
Table S1. Crystal Data and Structure Refinement for **3d**

Empirical formula	C ₂₃ H ₂₇ BrN ₄ O	
Formula weight	455.39	
Temperature	173.1500 K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 11.5777(16) Å	= 90°.
	b = 12.8278(18) Å	= 90°.
	c = 15.3442(19) Å	= 90°.
Volume	2169.6(5) Å ³	
Z	4	
Density (calculated)	1.394 Mg/m ³	

Absorption coefficient	1.916 mm ⁻¹
F(000)	944
Crystal size	0.528 x 0.436 x 0.206 mm ³
Theta range for data collection	1.906 to 27.506°.
Index ranges	-15<=h<=15, -16<=k<=15, -19<=l<=19
Reflections collected	18414
Independent reflections	9763 [R(int) = 0.0318]
Completeness to theta = 26.000°	98.3 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.00000 and 0.67856
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	9763 / 0 / 531
Goodness-of-fit on F ²	1.089
Final R indices [I>2sigma(I)]	R1 = 0.0496, wR2 = 0.1054
R indices (all data)	R1 = 0.0616, wR2 = 0.1122
Extinction coefficient	n/a
Largest diff. peak and hole	0.725 d -0.597 e.Å ⁻³

4. ¹H NMR, ¹³C NMR and HRMS of 3, 5

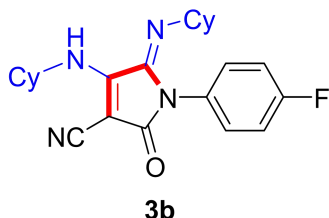
(Z)-4-(cyclohexylamino)-5-(cyclohexylimino)-2-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carbonitrile. (3a)



White solid, 189.6 mg, 84% yield, m.p. = 187 - 189 °C. ¹H NMR (CDCl₃, 500 MHz) δ 7.43 - 7.42 (t, *J* = 3.15 Hz, 3H, ArH), 7.24 - 7.21 (m, 2H, ArH), 6.68 (br s, 1H, NH), 3.39 (br s, 1H, CH), 3.00 - 2.95 (m, 1H, CH), 2.15 - 2.13 (m, 2H, CyH), 1.83-1.79 (m, 2H, CyH), 1.71 - 1.67 (m, 1H, CyH),

1.55 - 1.50 (m, 4H, CyH), 1.43 - 1.35 (m, 5H, CyH), 1.28 - 1.14 (m, 3H, CyH), 1.07 - 0.99 (m, 1H, CyH), 0.74 - 0.66 (m, 2H, Cy H); ¹³C NMR (CDCl₃, 125 MHz) δ 168.48, 153.51, 140.46, 135.20, 129.26, 129.04, 128.81, 114.05, 69.17, 55.67, 52.74, 33.62, 33.02, 25.11, 24.06; HRMS (ESI) *m/z* calcd for C₂₃H₂₉N₄O⁺ [M+H]⁺ 377.2336, found 377.2332.

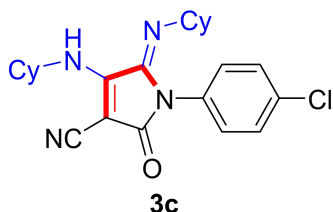
(Z)-4-(cyclohexylamino)-5-(cyclohexylimino)-1-(4-fluorophenyl)-2-oxo-2,5-dihydro-1H-pyrrole-3-carbonitrile. (3b (Z/E=11/89))



White solid, 193.9 mg, 82% yield, m.p. = 182 - 184 °C. ¹H NMR (CDCl₃, 500 MHz) δ 7.23 - 7.20 (m, 2H, ArH), 7.15 - 7.10 (m, 2H, ArH), 6.69 (br s, 1H, NH), 3.94 (br s, 1H, CH), 3.04 - 2.98 (m, 1H, CH), 2.15 - 2.12 (m, 2H, CyH), 1.83 - 1.79 (m, 2H, CyH), 1.71 - 1.68 (m, 1H, CyH),

1.60 - 1.56 (m, 2H, CyH), 1.50 - 1.39 (m, 7H, CyH), 1.26 - 1.18 (m, 3H, CyH), 1.11 - 1.03 (m, 1H, CyH), 0.81 - 0.73 (m, 2H, CyH); ¹³C NMR (CDCl₃, 125 MHz) δ 168.41, 162.66 (d, ¹*J*_{CF}=249.6 Hz), 153.46, 140.32, 131.16, 130.57 (d, ³*J*_{CF}=6.9 Hz), 116.26 (d, ²*J*_{CF}=22.8 Hz), 113.82, 69.23, 55.68, 52.74, 33.62, 33.00, 25.09, 24.02; HRMS (ESI) *m/z* calcd for C₂₃H₂₈FN₄O⁺ (M+H)⁺ 395.22417, found 395.22412.

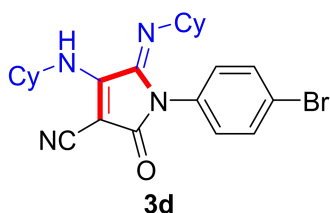
(Z)-1-(4-chlorophenyl)-4-(cyclohexylamino)-5-(cyclohexylimino)-2-oxo-2,5-dihydro-1H-pyrrole-3-carbonitrile. (3c (Z/E=8/92))



White solid, 216.6 mg, 88% yield, m.p. = 178 - 180 °C. ¹H NMR (CDCl₃, 500 MHz) δ 7.41 (d, *J* = 7.80 Hz, 2H, ArH), 7.17 (d, *J* = 7.85 Hz, 2H, ArH), 6.70 (br s, 1H, NH), 3.94 (br s, 1H, CH), 3.04 (br s, 1H, CH), 2.15 - 2.13 (m,

2H, CyH), 1.82 - 1.80 (m, 2H, CyH), 1.68 (br s, 1H, CyH), 1.59 - 1.57 (m, 2H, CyH), 1.50 - 1.41 (m, 7H, CyH), 1.26 - 1.21 (m, 3H, CyH), 1.09 - 1.07 (m, 1H, CyH), 0.80 - 0.77 (m, 2H, CyH); ^{13}C NMR (CDCl_3 , 125 MHz) δ 168.27, 153.45, 140.09, 135.00, 133.74, 130.03, 129.45, 113.82, 69.21, 55.79, 52.79, 33.57, 32.98, 25.16, 25.06, 24.02; **HRMS** (ESI) m/z calcd for $\text{C}_{23}\text{H}_{28}\text{ClN}_4\text{O}^+$ ($\text{M}+\text{H}$) $^+$ 411.19462, found 411.19455.

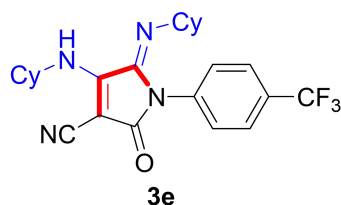
(Z)-1-(4-bromophenyl)-4-(cyclohexylamino)-5-(cyclohexylimino)-2-oxo-2,5-dihydro-1H-pyrrole-3-carbonitrile. (3d)



White solid, 234.3 mg, 86% yield, m.p. = 184 - 186 °C. ^1H NMR (CDCl_3 , 500 MHz) δ 7.55 (d, J = 8.45 Hz, 2H, ArH), 7.11 (d, J = 8.45 Hz, 2H, ArH), 6.68 (d, J = 7.65 Hz, 1H, NH), 3.93 - 3.92 (m, 1H, CH), 3.06 - 3.02 (m, 1H, CH), 2.14 - 2.12 (m, 2H, CyH), 1.82 - 1.79 (m, 2H, CyH), 1.70

- 1.68 (m, 1H, CyH), 1.58 - 1.57 (m, 2H, CyH), 1.51 - 1.35 (m, 7H, CyH), 1.27-1.17 (m, 3H, CyH), 1.10 - 1.03 (m, 1H, CyH), 0.81 - 0.74 (m, 2H, CyH); ^{13}C NMR (CDCl_3 , 125 MHz) δ 168.21, 153.47, 140.04, 134.28, 132.46, 130.34, 123.00, 113.82, 69.23, 55.83, 52.80, 33.58, 33.00, 25.19, 25.08, 24.04; **HRMS** (ESI) m/z calcd for $\text{C}_{23}\text{H}_{28}\text{BrN}_4\text{O}^+$ ($\text{M}+\text{H}$) $^+$ 455.14410, found 455.14386.

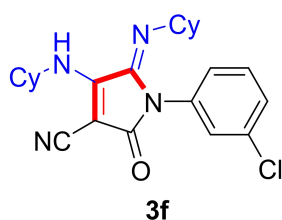
(Z)-4-(cyclohexylamino)-5-(cyclohexylimino)-2-oxo-1-(4-(trifluoromethyl)phenyl)-2,5-dihydro-1H-pyrrole-3-carbonitrile. (3e (Z/E=8/92))



White solid, 242.5 mg, 91% yield, m.p. = 193 - 195 °C. ^1H NMR (CDCl_3 , 500 MHz) δ 7.71 (d, J = 8.0 Hz, 2H, ArH), 7.38 (d, J = 7.9 Hz, 2H, ArH), 6.72 (d, J = 6.8 Hz, 1H, NH), 3.94 (br s, 1H, CH), 2.98 - 2.94 (m, 1H, CH), 2.15 - 2.13 (m, 2H, CyH), 1.83 - 1.80 (m, 2H, CyH), 1.71

- 1.69 (m, 1H, CyH), 1.58 - 1.53 (m, 2H, CyH), 1.50 - 1.40 (m, 7H, CyH), 1.29 - 1.20 (m, 3H, CyH), 1.08 - 1.03 (m, 1H, CyH), 0.76 - 0.68 (m, 2H, CyH); ^{13}C NMR (CDCl_3 , 125 MHz) δ 167.99, 153.49, 139.86, 138.62, 131.08 (q, $^2J_{\text{CF}}=32.91$ Hz), 129.17, 126.29, 123.57 (q, $^1J_{\text{CF}}=271.93$ Hz), 113.70, 69.29, 56.15, 52.88, 33.49, 32.98, 25.07, 23.99; **HRMS** (ESI) m/z calcd for $\text{C}_{24}\text{H}_{28}\text{F}_3\text{N}_4\text{O}^+$ ($\text{M}+\text{H}$) $^+$ 445.22097, found 445.22110.

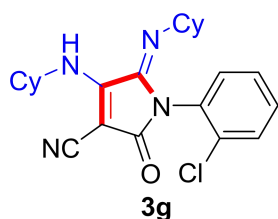
(Z)-1-(3-chlorophenyl)-4-(cyclohexylamino)-5-(cyclohexylimino)-2-oxo-2,5-dihydro-1H-pyrrole-3-carbonitrile. (3f)



White solid, 196.8 mg, 80% yield, m.p. = 167 - 169 °C. ^1H NMR (CDCl_3 , 500 MHz) δ 7.43 - 7.36 (m, 2H, ArH), 7.26 (s,

1H, ArH), 7.16 (d, $J = 7.7$ Hz, 1H, ArH), 6.70 (d, $J = 7.8$ Hz, 1H, NH), 3.97 - 3.91 (br s, 1H, CH), 3.02 - 2.97 (br s, 1H, CH), 2.15 - 2.12 (m, 2H, CyH), 1.83 - 1.80 (m, 2H, CyH), 1.71 - 1.69 (m, 1H, CyH), 1.60 - 1.58 (m, 2H, CyH), 1.53 - 1.36 (m, 7H, CyH), 1.29 - 1.20 (m, 3H, CyH), 1.11 - 1.04 (m, 1H, CyH), 0.79 - 0.76 (m, 2H, CyH); ^{13}C NMR (CDCl₃, 125 MHz) δ 168.03, 153.46, 140.02, 136.45, 134.78, 130.10, 129.19, 128.96, 127.01, 113.73, 69.31, 56.03, 52.79, 33.56, 32.99, 25.19, 25.07, 24.02; HRMS (ESI) m/z calcd for C₂₃H₂₈ClN₄O⁺ (M+H)⁺ 411.19462, found 411.19455.

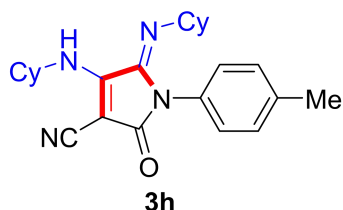
(Z)-1-(2-chlorophenyl)-4-(cyclohexylamino)-5-(cyclohexylimino)-2-oxo-2,5-dihydro-1H-pyrrole-3-carbonitrile. (3g)



White solid, 204.3 mg, 83% yield, m.p. = 161 - 163 °C. IR (KBr) ν : 3435, 3264, 2934, 2854, 2212, 1735, 1722, 1681, 1640, 1530, 1481, 1449, 1370, 1089; ^1H NMR (CDCl₃, 500 MHz) δ 7.49 (d, $J = 7.8$ Hz, 1H, ArH), 7.41 - 7.39 (m, 1H, ArH), 7.35 (d, $J = 3.3$ Hz, 2H, ArH), 6.68 (br s, 1H, NH), 3.95

(br s, 1H, CH), 2.88 (br s, 1H, CH), 2.17 (m, 2H, CyH), 1.83 - 1.81 (m, 2H, CyH), 1.72 - 1.69 (m, 1H, CyH), 1.60 - 1.39 (m, 8H, CyH), 1.34 - 1.14 (m, 4H, CyH), 1.06 - 1.04 (m, 1H, CyH), 0.80 - 0.65 (m, 2H, CyH); ^{13}C NMR (CDCl₃, 125 MHz) δ 167.68, 153.43, 139.50, 134.37, 133.20, 131.27, 130.68, 129.95, 127.67, 113.89, 69.33, 55.99, 52.83, 34.10, 33.61, 33.07, 25.10, 24.09; HRMS (ESI) m/z calcd for C₂₃H₂₈ClN₄O⁺ (M+H)⁺ 411.19462, found 411.19452.

(Z)-4-(cyclohexylamino)-5-(cyclohexylimino)-2-oxo-1-(p-tolyl)-2,5-dihydro-1H-pyrrole-3-carbonitrile. (3h)

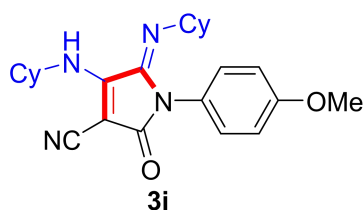


White solid, 166.2 mg, 71% yield, m.p. = 179 - 183 °C. IR (KBr) ν : 3435, 3231, 2934, 2854, 2209, 1718, 1675, 1638, 1620, 1534, 1512, 1449, 1362, 1070, 805 cm⁻¹; ^1H NMR (CDCl₃, 500 MHz) δ 7.22 (d, $J = 7.8$ Hz, 2H, ArH), 7.10 (d, $J = 7.9$ Hz, 2H, ArH), 6.69 (br s, 1H, NH),

3.93 (br s, 1H, CH), 3.08 - 3.04 (br s, 1H, CH), 2.40(s, 3H, CH), 2.15 - 2.13 (m, 2H, CyH), 1.82 - 1.80 (m, 2H, CyH), 1.71 - 1.68 (m, 1H, CyH), 1.56 - 1.36 (m, 9H, CyH), 1.28 - 1.16 (m, 3H, CyH), 1.07 - 1.04 (m, 1H, CyH), 0.78 - 0.70 (m, 2H, CyH); ^{13}C NMR (CDCl₃, 125 MHz) δ 168.66, 153.51, 140.58, 139.09, 132.42, 129.85, 128.50, 114.10, 69.12, 55.43, 52.69, 33.63, 32.98, 25.22, 24.04, 21.20; HRMS (ESI) m/z calcd for C₂₄H₃₁N₄O⁺ (M+H)⁺ 391.24924, found 391.24933.

(Z)-4-(cyclohexylamino)-5-(cyclohexylimino)-1-(4-methoxyphenyl)-2-oxo-2,5-dihydro-1H-pyrrole-3-carbonitrile. (3i)

ydro-1H-pyrrole-3-carbonitrile. (3i)

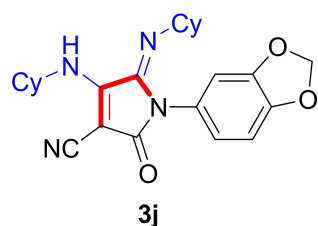


White solid, 143.8 mg, 59% yield, m.p. = 182 - 183 °C.

¹H NMR (CDCl₃, 500 MHz) δ 7.14 (d, *J* = 8.55 Hz, 2H, ArH), 6.93 (d, *J* = 8.55 Hz, 2H, ArH), 6.69 (br s, 1H, NH), 3.93 (br s, 1H, CH), 3.84 (s, 3H, CH), 3.07 (m, 1H, CH), 2.15 -2.05 (m, 2H, CyH), 1.81 (m, 2H,

CyH), 1.67 (m, 2H, CyH), 1.55 (s, 1H, CyH), 1.39 - 1.50 (m, 7H, CyH), 1.19 - 1.27 (m, 3H, CyH), 1.02 - 1.10 (m, 1H, CyH), 0.73 - 0.81 (m, 2H, CyH); ¹³C NMR (CDCl₃, 125 MHz) δ 168.80, 159.92, 153.46, 140.65, 129.87, 127.53, 114.51, 114.11, 69.05, 55.54, 55.38, 52.69, 33.67, 33.00, 25.21, 25.09, 24.06; HRMS (ESI) m/z calcd for C₂₄H₃₁N₄O₂⁺(M+H)⁺407.24415, found 407.24414.

(Z)-1-(benzo[d][1,3]dioxol-5-yl)-4-(cyclohexylamino)-5-(cyclohexylimino)-2-oxo-2,5-dihydro-1H-pyrrole-3-carbonitrile. (3j)

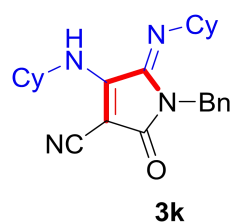


White solid, 133.6 mg, 53% yield, m.p. = 181 - 184 °C. ¹H

NMR (CDCl₃, 500 MHz) δ 6.84 (d, *J* = 8.40 Hz, 1H, NH), 6.72 - 6.71 (m, 3H, ArH), 6.05 (d, *J* = 18.70 Hz, 2H, CH₂), 3.93 (br s, 1H, CH), 3.16 (br s, 1H, CH), 2.15 (d, *J* = 10.35 Hz, 2H, CyH), 1.82 (d, *J* = 13.20 Hz, 2H, CyH), 1.71 (d, *J*

= 12.85 Hz, 1H,CyH), 1.61 (s, 2H, CyH), 1.51 - 1.39 (m, 7H, CyH), 1.27 - 1.23 (m, 3H, CyH), 1.12 - 1.09 (m, 1H, CyH), 0.88 - 0.83 (m, 2H, CyH); ¹³C NMR (CDCl₃, 125 MHz) δ 168.68, 153.40, 148.20, 148.16, 140.45, 128.51, 122.44, 114.05, 109.58, 108.32, 101.93, 68.06, 55.52, 55.73, 33.65, 33.00, 25.25, 25.08, 24.07; HRMS (ESI) m/z calcd for C₂₄H₂₉N₄O₃⁺(M+H)⁺ 421.22342, found 421.22342.

(Z)-1-benzyl-4-(cyclohexylamino)-5-(cyclohexylimino)-2-oxo-2,5-dihydro-1H-pyrrole-3-carbonitrile. (3k)



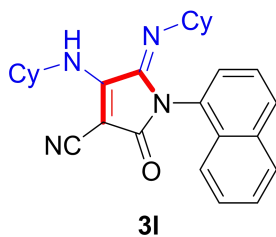
White solid, 173.3 mg, 74% yield, m.p. = 158 - 161 °C. ¹H NMR

(CDCl₃, 500 MHz) δ 7.32 (t, *J* = 7.43 Hz, 2H, ArH), 7.25 (t, *J* = 7.37 Hz, 1H, ArH), 7.10 (d, *J* = 7.35 Hz, 2H, ArH), 6.68 (br s, 1H, NH), 4.91 (s, 2H, CH), 3.90 (br s, 1H, CH), 3.66 - 3.62 (m, 1H, CH), 2.12 (d, 2H, CyH), 1.81 - 1.78 (m, 2H, CyH), 1.70 - 1.67 (m,

1H, CyH), 1.63 - 1.67 (m, 2H, CyH), 1.56 - 1.53 (m, 1H, CyH), 1.49 - 1.43 (m, 2H, CyH), 1.40 - 1.35 (m, 2H, CyH), 1.32 - 1.21 (m, 5H, CyH), 1.15 - 0.99 (m, 3H, CyH); ¹³C NMR (CDCl₃, 125 MHz) δ 169.73, 153.18, 140.90, 114.22, 69.34, 56.09, 52.36, 34.74, 32.91, 30.47, 29.65, 26.65, 25.43, 25.10, 24.96, 24.38, 23.98; HRMS (ESI)

m/z calcd for C₂₄H₃₁N₄O⁺(M+H)⁺ 391.24924, found 391.24924.

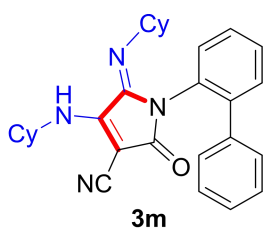
(Z)-4-(cyclohexylamino)-5-(cyclohexylimino)-1-(2,3-dihydronaphthalen-1-yl)-2-oxo-2,5-dihydro-1H-pyrrole-3-carbonitrile. (3l (Z/E=8/92))



3l

White solid, 225.1 mg, 88% yield, m.p. = 219 - 222 °C. ¹H NMR (CDCl₃, 500 MHz) δ 7.96 - 7.90 (m, 2H, ArH), 7.63 - 7.62 (m, 1H, ArH), 7.54 - 7.49 (m, 3H, ArH), 7.42 (d, *J* = 7.1 Hz, 1H, ArH), 6.76 (br s, 1H, NH), 4.01 (br s, 1H, CH), 2.60 - 2.56 (br s, 1H, CH), 2.21 (br s, 2H, CyH), 1.84 (br s, 2H, CyH), 1.73 - 1.71 (m, 1H, CyH), 1.56 - 1.42 (m, 5H, CyH), 1.31 - 1.23 (m, 4H, CyH), 1.13 - 0.98 (m, 2H, CyH), 0.93 - 0.86 (m, 2H, CyH), 0.44 - 0.48(m, 1H, CyH), 0.12 - 0.15(m, 1H, CyH); ¹³C NMR (CDCl₃, 125 MHz) δ 168.43, 153.46, 140.68, 134.09, 131.71, 131.38, 129.77, 128.45, 127.43, 127.08, 126.75, 125.28, 122.16, 114.06, 69.30, 55.99, 52.86, 33.99, 33.56, 33.08, 25.00, 24.08, 23.72; HRMS (ESI) m/z calcd for C₂₇H₃₁N₄O⁺ (M+H)⁺ 427.24924, found 427.24893.

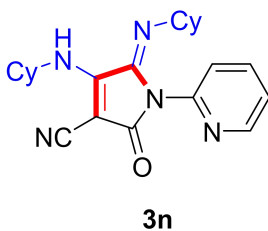
(Z)-1-([1,1'-biphenyl]-2-yl)-4-(cyclohexylamino)-5-(cyclohexylimino)-2-oxo-2,5-dihydro-1H-pyrrole-3-carbonitrile. (3m)



3m

White solid, 236.1 mg, 87% yield, m.p. = 176 - 178 °C. ¹H NMR (CDCl₃, 500 MHz) δ 7.51 (t, *J* = 7.5 Hz, 1H, ArH), 7.42 (t, *J* = 7.3 Hz, 2H, ArH), 7.38 - 7.28 (m, 4H, ArH), 7.22 (d, *J* = 7.0 Hz, 2H, ArH), 6.41 (br s, 1H, NH), 3.84 (br s, 1H, CH), 2.89 - 2.84 (m, 1H, CH), 2.08 - 2.01 (m, 2H, CyH), 1.77 - 1.75 (m, 2H, CyH), 1.67 - 1.59 (m, 2H, CyH), 1.48 - 1.43 (m, 4H, CyH), 1.34 - 1.10 (m, 6H, CyH), 1.05 - 0.98 (m, 2H, CyH), 0.94 - 0.86 (m, 1H, CyH), 0.61 - 0.53 (m, 1H, CyH); ¹³C NMR (CDCl₃, 125 MHz) δ 163.31, 153.52, 142.35, 139.95, 138.19, 132.91, 130.57, 129.96, 129.76, 128.46, 128.33, 127.78, 114.16, 68.89, 55.35, 52.62, 34.22, 33.32, 32.83, 25.07, 24.00; HRMS (ESI) m/z calcd for C₂₉H₃₃N₄O⁺ (M+H)⁺ 453.26489, found 453.26456.

(Z)-4-(cyclohexylamino)-5-(cyclohexylimino)-2-oxo-1-(pyridin-2-yl)-2,5-dihydro-1H-pyrrole-3-carbonitrile. (3n (Z/E=26/74))

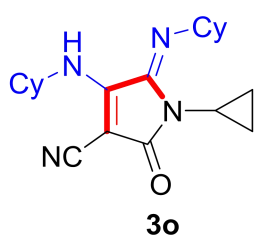


3n

White solid, 178.8 mg, 79% yield, m.p. = 143 - 145 °C. ¹H NMR (CDCl₃, 500 MHz) δ 8.50 (d, *J* = 3.6 Hz, 1H, ArH), 7.84 (t, *J* = 7.6 Hz, 1H, ArH), 7.38 (d, *J* = 7.8 Hz, 1H, ArH), 7.34 (t, *J* = 7.0 Hz, 1H, ArH), 6.75 (br s, 1H, NH), 3.94 (br s, 1H, CH), 2.86 - 2.82 (m, 1H, CH), 2.15 - 2.12 (m, 2H, CyH),

1.94 - 1.92 (m, 1H, CyH), 1.82 - 1.80 (m, 2H, CyH), 1.70 - 1.68 (m, 2H, CyH), 1.60 - 1.33 (m, 7H, CyH), 1.31 - 1.22 (m, 3H, CyH), 1.16 - 1.05 (m, 1H, CyH), 0.81 - 0.74 (m, 2H, CyH); ^{13}C NMR (CDCl₃, 125 MHz) δ 168.15, 153.88, 148.92, 148.78, 139.92, 138.32, 123.53, 123.32, 113.87, 69.44, 57.57, 52.79, 33.27, 32.97, 25.28, 25.06, 24.91, 24.03; HRMS (ESI) m/z calcd for C₂₂H₂₈N₅O⁺ (M+H)⁺ 378.22884, found 378.22888;

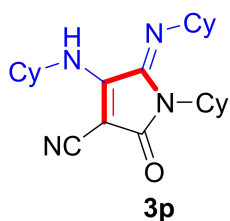
(Z)-4-(cyclohexylamino)-5-(cyclohexylimino)-1-cyclopropyl-2-oxo-2,5-dihydro-1H-pyrrole-3-carbonitrile. (3o)



White solid, 136.8 mg, 67% yield, m.p. = 125 - 128 °C. ^1H NMR (CDCl₃, 500 MHz) δ 6.59 (d, J = 5.09 Hz, 1H, NH), 4.55 - 4.52 (m, 1H, CH), 3.81 (br s, 1H, CH), 2.62 (m, 1H, CH), 2.05 (m, 2H, CH), 1.81 - 1.75 (m, 4H, CH), 1.71 - 1.65 (m, 5H, CH), 1.48 - 1.40 (m, 3H, CH), 1.36 - 1.28 (m, 5H, CH), 1.25 -

1.23 (m, 1H, CH), 0.99 - 0.95 (m, 4H, CH); ^{13}C NMR (CDCl₃, 125 MHz) δ 169.02, 152.94, 141.27, 114.16, 68.94, 55.86, 52.49, 34.69, 32.92, 25.49, 25.08, 24.33, 23.99, 9.16; HRMS (ESI) m/z calcd for C₂₀H₂₉N₄O⁺(M+H)⁺ 341.23359, found 341.23364.

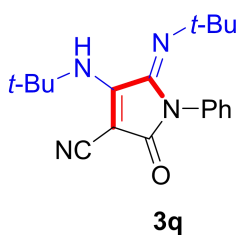
(Z)-1-cyclohexyl-4-(cyclohexylamino)-5-(cyclohexylimino)-2-oxo-2,5-dihydro-1H-pyrrole-3-carbonitrile. (3p)



White solid, 160.5 mg, 70% yield, m.p. = 111 - 113 °C. ^1H NMR (CDCl₃, 500 MHz) δ 6.61 (br s, 1H, NH), 3.81 - 3.75 (m, 3H, CH), 2.33 - 2.23(s, 2H, CyH), 2.07 - 2.05 (m, 2H, CyH), 1.88 - 1.81 (m, 5H, CyH), 1.75 - 1.70 (m, 7H, CyH), 1.46 - 1.42 (m, 4H, CyH), 1.37 - 1.30 (m, 5H, CyH), 1.25 - 1.16 (m, 5H, CyH); ^{13}C

NMR (CDCl₃, 125 MHz) δ 169.22, 153.60, 140.49, 137.19, 128.80, 127.48, 125.78, 114.20, 68.55, 56.11, 52.75, 44.46, 34.44, 32.97, 25.29, 25.08, 24.06; HRMS (ESI) m/z calcd for C₂₃H₃₅N₄O⁺(M+H)⁺ 383.28054, found 383.28058.

(Z)-4-(tert-butylamino)-5-(tert-butylimino)-2-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carbonitrile. (3q)

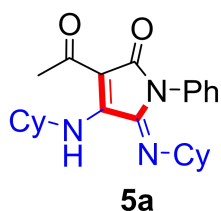


White solid, 116.7 mg, 60% yield, m.p. = 211 - 212 °C. ^1H NMR (CDCl₃, 500 MHz) δ 7.44 - 7.43 (m, 3H, ArH), 7.37 (br s, 1H, NH), 7.21 - 7.20 (m, 2H, ArH), 1.57 (s, 9H, *t*-Bu-H), 0.98 (s, 9H, *t*-Bu-H); ^{13}C NMR (CDCl₃, 125 MHz) δ 170.56, 152.09, 139.30, 136.49, 130.26, 129.13, 116.02, 68.94, 54.69, 53.18,

31.08, 29.71; HRMS (ESI) m/z calcd for C₁₉H₂₅N₄O⁺(M+H)⁺ 325.20229, found

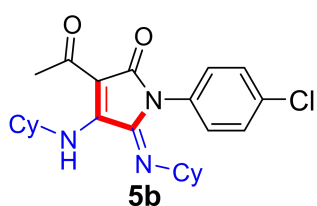
325.20221.

(Z)-3-acetyl-4-(cyclohexylamino)-5-(cyclohexylimino)-1-phenyl-1,5-dihydro-2H-pyrrol-2-one. (5a)



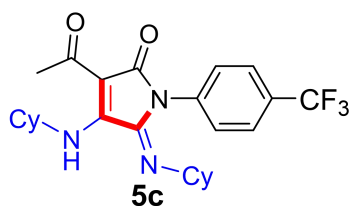
White solid, 160.4 mg, 68% yield, m.p. = 199 – 201 °C. ¹H NMR (CDCl₃, 500 MHz) δ 10.37 (d, *J* = 6.10 Hz, 1H, NH), 7.43 - 7.40 (m, 3H, ArH), 7.26 (d, *J* = 8.00 Hz, 2H, ArH), 4.90 (br s, 1H, CH), 3.10 - 3.06 (m, 1H, CH), 2.48 (s, 3H, CH₃), 2.04 (s, 2H, CyH), 1.78 (s, 2H, CyH), 1.63 (d, 1H, CyH), 1.57 - 1.54 (m, 2H, CyH), 1.47 - 1.45 (m, 2H, CyH), 1.40 - 1.32 (m, 5H, CyH), 1.26 - 1.18 (m, 3H, CyH), 1.15 - 1.08 (m, 1H, CyH), 0.82 - 0.75 (m, 2H, CyH); ¹³C NMR (CDCl₃, 125 MHz) δ 195.41, 169.47, 158.10, 140.88, 136.57, 129.07, 128.78, 128.46, 98.34, 56.28, 52.33, 33.64, 33.26, 28.04, 25.32, 24.44, 23.82; HRMS (ESI) m/z calcd for C₂₄H₃₂N₃O₂⁺ (M+H)⁺ 394.24890, found 394.24881.

(Z)-3-acetyl-1-(4-chlorophenyl)-4-(cyclohexylamino)-5-(cyclohexylimino)-1,5-dihydro-2H-pyrrol-2-one. (5b)



White solid, 64.1 mg, 25% yield, m.p. = 182 - 184 °C. ¹H NMR (CDCl₃, 500 MHz) δ 10.39 (d, *J* = 7.45 Hz, 1H, NH), 7.43 -7.41 (m, 2H, ArH), 7.22 - 7.19 (m, 2H, ArH), 4.90 - 4.89 (m, 1H, CH), 3.16 - 3.12 (m, 1H, CH), 2.48 (s, 3H, CH₃), 2.04 (br s, 2H, CyH), 1.80 - 1.79 (m, 2H, CyH), 1.62 - 1.59 (m, 4H, CyH), 1.50 - 1.47 (m, 2H, CyH), 1.38 - 1.35 (m, 4H, CyH), 1.30 - 1.25 (m, 3H, CyH), 1.18 - 1.13 (m, 1H, CyH), 0.91 - 0.86 (m, 2H, CyH); ¹³C NMR (CDCl₃, 125 MHz) δ 195.41, 169.22, 158.02, 140.58, 135.14, 134.33, 129.99, 129.26, 98.25, 56.38, 52.41, 33.58, 33.26, 28.09, 25.27, 24.44, 23.75; HRMS (ESI) m/z calcd for C₂₄H₃₁ClN₃O₂⁺(M+H)⁺ 428.20993, found 428.21002.

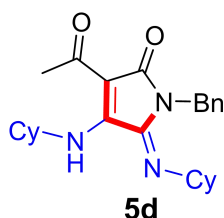
(Z)-3-acetyl-4-(cyclohexylamino)-5-(cyclohexylimino)-1-(4-(trifluoromethyl)phenyl)-1,5-dihydro-2H-pyrrol-2-one. (5c)



White solid, 44.3 mg, 16% yield, m.p. = 134 - 138 °C. ¹H NMR (CDCl₃, 500 MHz) δ 10.40 (d, *J* = 7.75 Hz, 1H, NH), 7.71 (d, *J* = 8.25 Hz, 2H, ArH), 7.40 (d, *J* = 8.20 Hz, 2H, ArH), 4.88 (m, 1H, CH), 3.07 - 3.03 (m, 1H, CH), 2.48 (s, 3H, CH₃), 2.04 (br s, 2H, CyH), 1.80 (br s, 2H, CyH), 1.62 - 1.57 (m, 4H, CyH), 1.48 - 1.46 (m, 2H, CyH), 1.39 - 1.35 (m, 4H, CyH), 1.29 - 1.25 (m, 3H, CyH), 1.16 - 1.14 (m, 1H, CyH), 0.82 - 0.79 (m, 2H,

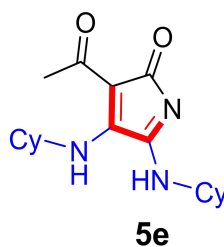
CyH); ^{13}C NMR (CDCl_3 , 125 MHz) δ 195.42, 168.87, 158.05, 140.47, 140.06, 130.47(q, $^2J_{\text{CF}}=32.96$ Hz), 128.99, 126.06, 123.72 (q, $^1J_{\text{CF}}=272.27$ Hz), 98.28, 56.82, 52.47, 33.45, 33.27 28.08, 25.26, 24.41, 23.70; **HRMS** (ESI) m/z calcd for $\text{C}_{25}\text{H}_{31}\text{F}_3\text{N}_3\text{O}_2^+$ (M+H) $^+$ 462.23629, found 462.23633

(Z)-3-acetyl-1-benzyl-4-(cyclohexylamino)-5-(cyclohexylimino)-1,5-dihydro-2H-pyrrol-2-one. (5d)



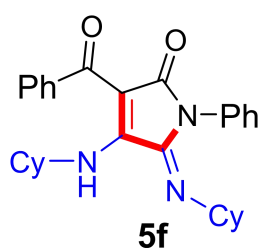
White solid, 102.6 mg, 42% yield, m.p. = 170 - 172 °C. ^1H NMR (CDCl_3 , 500 MHz) δ 10.39 (d, $J = 6.47$ Hz, 1H, NH), 7.33 (t, $J = 7.53$ Hz, 2H, ArH), 7.24 (t, $J = 7.30$ Hz, 1H, ArH), 7.12 (d, $J = 7.45$ Hz, 2H, ArH), 4.94 (s, 2H, CH_2), 4.85 (br s, 1H, CH), 3.77 - 3.73 (m, 1H, CH), 2.53 (s, 3H, CH_3), 1.99 (br s, 2H, CH), 1.75 - 1.69 (m, 3H, CyH), 1.65 - 1.62 (m, 3H, CyH), 1.55 - 1.53 (m, 1H, CyH), 1.41 - 1.39 (m, 2H, CyH), 1.32 - 1.26 (m, 6H, CyH), 1.19 - 1.15 (m, 1H, CyH), 1.11 - 1.06 (m, 2H, CyH); ^{13}C NMR (CDCl_3 , 125 MHz) δ 195.10, 169.70, 153.19, 140.85, 138.00, 128.70, 127.14, 125.67, 98.16, 56.57, 52.17, 44.16, 34.72, 33.22, 28.01, 25.42, 25.26, 24.42, 23.91; **HRMS** (ESI) m/z calcd for $\text{C}_{25}\text{H}_{34}\text{N}_3\text{O}_2^+$ (M+H) $^+$ 408.26455, found 408.26459.

3-acetyl-4,5-bis(cyclohexylamino)-2H-pyrrol-2-one (5e)



White solid; 57.1 mg, 30% yield, m.p. = 218 - 219 °C; ^1H NMR (CDCl_3 , 500 MHz) δ 10.18 (d, $J = 7.50$ Hz, 1H, NH), 8.95 (s, 1H, NH), 4.88 - 4.86 (m, 1H, CH), 3.43 (br s, 1H, CH), 2.48 (s, 3H, CH_3), 2.03 - 2.01 (m, 2H, CH), 1.79 - 1.77 (m, 5H, CyH), 1.65 - 1.62 (m, 3H, CyH), 1.48 - 1.42 (m, 4H, CyH), 1.39 - 1.32 (m, 5H, CyH), 1.30 - 1.25 (m, 1H, CyH); ^{13}C NMR (CDCl_3 , 125 MHz) δ 195.01, 170.78, 158.15, 144.43, 99.36, 58.74, 52.49, 33.24, 33.13, 27.97, 25.67, 25.24, 24.37, 24.04; **HRMS** (ESI) m/z calcd for $\text{C}_{18}\text{H}_{28}\text{N}_3\text{O}_2^+$ (M+H) $^+$ 318.21760, found 318.21762.

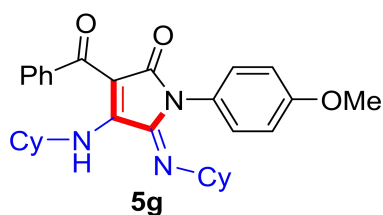
(Z)-3-benzoyl-4-(cyclohexylamino)-5-(cyclohexylimino)-1-phenyl-1,5-dihydro-2H-pyrrol-2-one (5f)



White solid, 60.3 mg, 21% yield, m.p. = 211-213°C. ^1H NMR (CDCl_3 , 500 MHz) δ 10.90 (d, $J = 6.60$ Hz, 1H, NH), 7.71 (d, $J = 7.45$ Hz, 2H, ArH), 7.41 - 7.34 (m, 6H, ArH), 7.25 (d, $J = 7.15$ Hz, 2H, ArH), 5.02 - 5.00 (m, 1H, CH), 3.12 - 3.09 (m, 1H, CH), 2.12 - 2.10 (m, 2H, CyH), 1.83 - 1.81 (m, 2H, CyH), 1.66 - 1.64 (m, 1H, CyH), 1.58 - 1.56 (m, 2H, CyH), 1.50 - 1.48 (m,

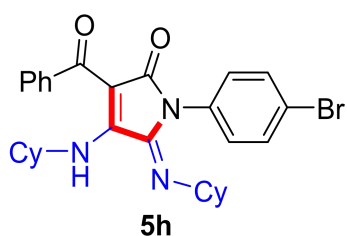
2H, CyH), 1.44 - 1.38 (m, 5H, CyH), 1.28 - 1.22 (m, 3H, CyH), 1.16 - 1.09 (m, 1H, CyH), 0.83 - 0.78 (m, 2H, CyH), ^{13}C NMR (CDCl₃, 125 MHz) δ 191.82, 168.21, 160.02, 140.73, 139.27, 136.52, 130.78, 129.00, 128.84, 128.49, 127.30, 97.51, 56.31, 52.62, 33.69, 33.27, 25.32, 24.45, 23.87, HRMS (ESI) m/z calcd for C₂₉H₃₃N₃O₂⁺ (M+Na)⁺ 478.2470, found 478.2474.

(Z)-3-benzoyl-4-(cyclohexylamino)-5-(cyclohexylimino)-1-(4-methoxyphenyl)-1,5-dihydro-2H-pyrrol-2-one (5g)



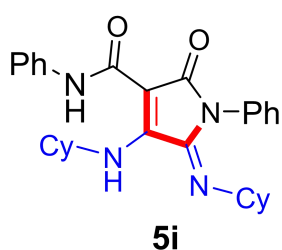
White solid, 66.9 mg, 23% yield, m.p. =198 -200 °C. ^1H NMR (CDCl₃, 500 MHz) δ 10.91 (d, J = 7.15 Hz, 1H, NH), 7.71 (d, J = 7.30 Hz, 2H, ArH), 7.40 (d, J = 7.25 Hz, 1H, ArH), 7.36 - 7.33 (m, 2H, ArH), 7.16 (d, J = 8.70 Hz, 2H, ArH), 6.91 (d, J = 8.55 Hz, 2H, ArH), 5.02 (br s, 1H, CH), 3.81 (s, 3H, MeH) 3.23 - 3.19 (m, 1H, CH), 2.11 - 2.10 (m, 2H, CyH), 1.82 - 1.80 (m, 2H, CyH), 1.66 - 1.58 (m, 3H, CyH), 1.51 - 1.49 (m, 2H, CyH), 1.44 - 1.38 (m, 5H, CyH), 1.27 - 1.23 (m, 3H, CyH), 1.16 - 1.12 (m, 1H, CyH), 0.90 - 0.83 (m, 2H, CyH); ^{13}C NMR (CDCl₃, 125 MHz) δ 191.73, 168.50, 160.00, 159.51, 140.92, 139.27, 130.75, 129.88, 128.45, 128.97, 128.55, 127.73, 127.27, 114.30, 97.48, 55.96, 55.52, 52.56, 33.76, 33.28, 25.32, 24.45, 23.87; HRMS (ESI) m/z calcd for C₃₀H₃₆N₃O₃⁺ (M+H)⁺ 486.27512, found 486.27499

(Z)-3-benzoyl-1-(4-bromophenyl)-4-(cyclohexylamino)-5-(cyclohexylimino)-1,5-dihydro-2H-pyrrol-2-one (5h)



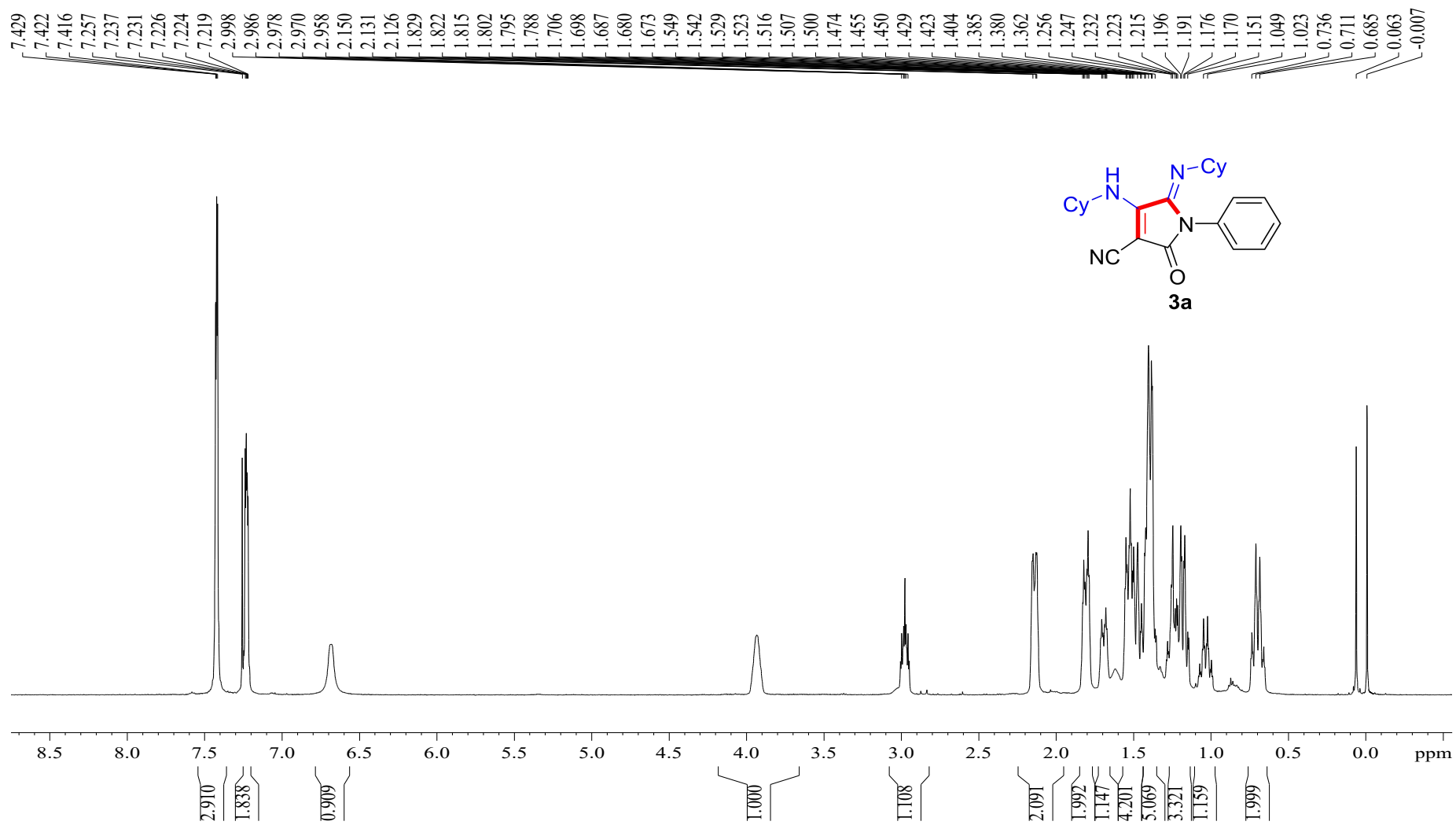
White solid, 38.4 mg, 12% yield, m.p. =203 - 205°C. ^1H NMR (CDCl₃, 500 MHz) δ 10.89 (d, 1H, NH), 7.78 - 7.69 (d, 2H, ArH), 7.53 - 7.52 (d, 2H, ArH), 7.48-7.34 (m, 3H, ArH), 7.14 - 7.12 (d, 2H, ArH), 5.00 (d, 1H, CH), 3.19 - 3.11 (m, 1H, CH), 2.10 - 2.09 (d, 2H, CyH), 1.94 - 1.80 (d, 2H, CyH), 1.66 - 1.61 (m, 3H, CyH), 1.51 - 1.48 (m, 3H, CyH), 1.35 - 1.31 (m, 4H, CyH), 1.29 - 1.21 (m, 3H, CyH), 1.18 - 0.92 (m, 1H, CyH), 0.89 - 0.84(m, 2H, CyH); ^{13}C NMR (CDCl₃, 125 MHz) δ 191.82, 167.91, 159.90, 140.36, 139.15, 135.63, 132.15, 130.90, 130.35, 129.40, 128.45, 127.81, 127.34, 122.27, 97.38, 56.43, 52.68, 33.60, 33.26, 25.29, 24.43, 23.76; HRMS (ESI) m/z calcd for C₂₉H₃₃BrN₃O₂⁺ (M+H)⁺ 534.1756, found 534.1750.

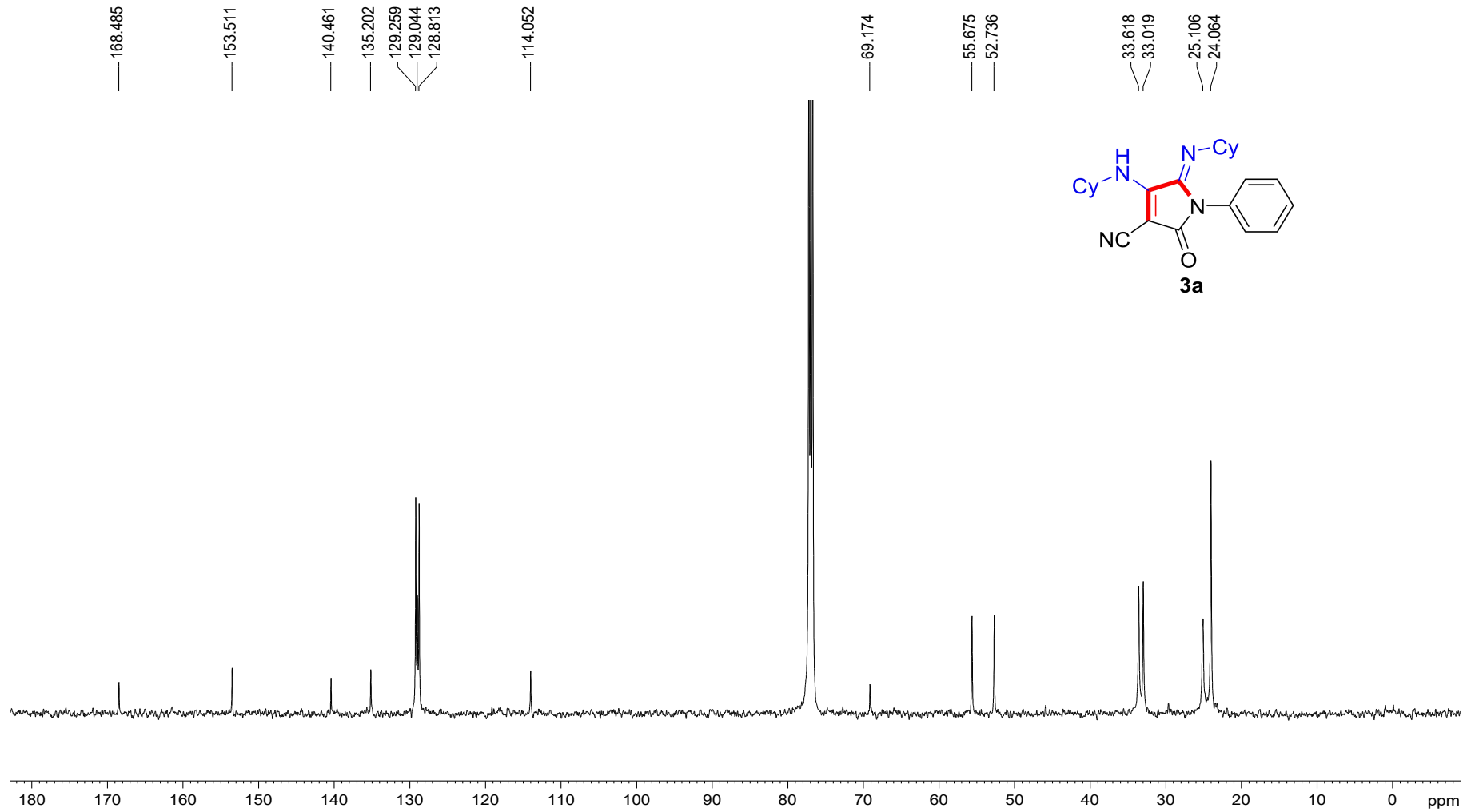
(Z)-4-(cyclohexylamino)-5-(cyclohexylimino)-2-oxo-N,1-diphenyl-2,5-dihydro-1H-pyrrole-3-carboxamide (5i)



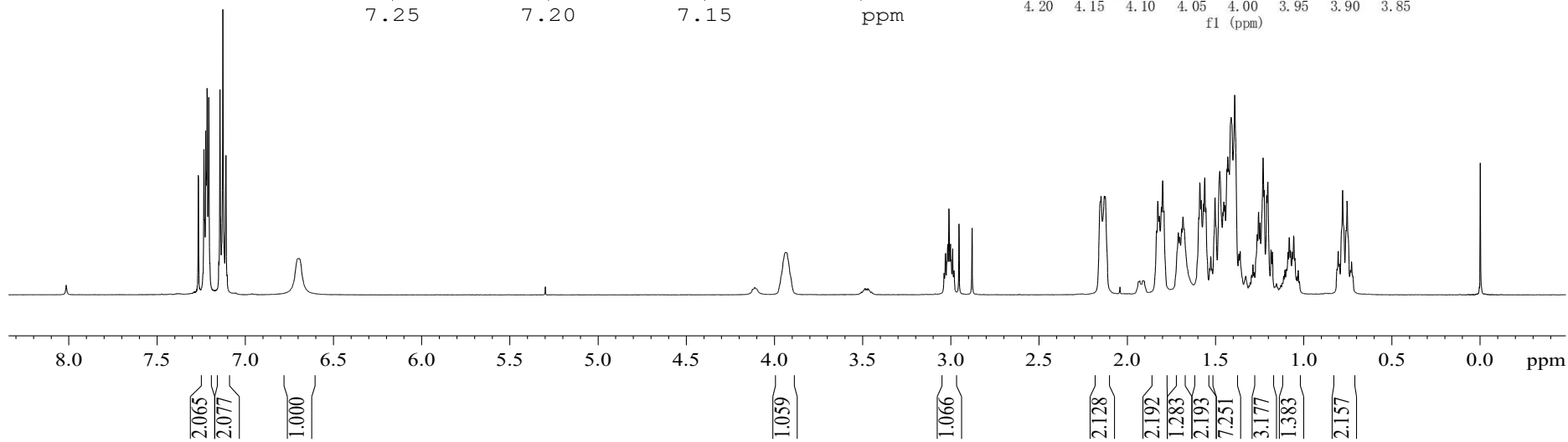
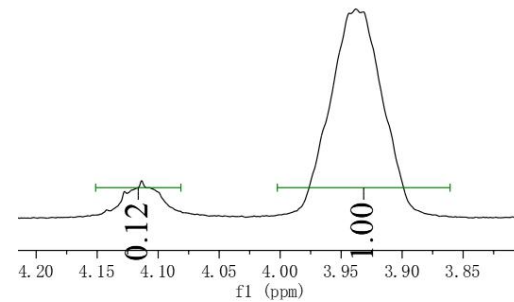
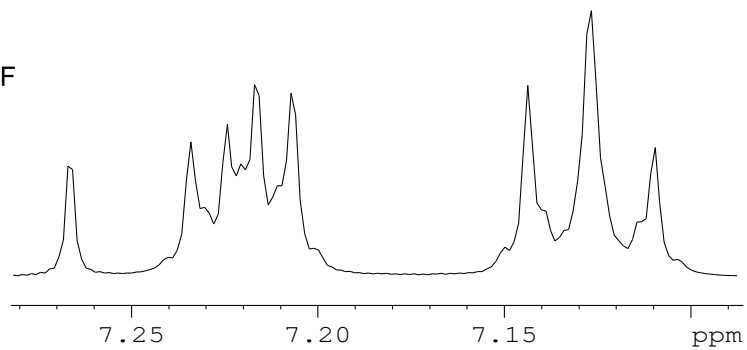
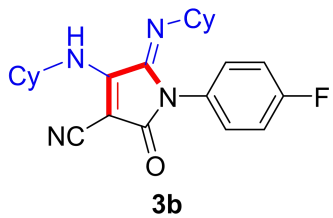
White solid, 87.5 mg, 31% yield, m.p. = 211- 213°C. ^1H NMR (CDCl₃, 500 MHz) δ 9.81 (s, 1H, NH), 9.29 (s, 1H, NH), 7.55 - 7.44 (d, 5H, ArH), 7.28 (s, 4H, ArH), 7.04 (s, 1H, ArH), 4.81

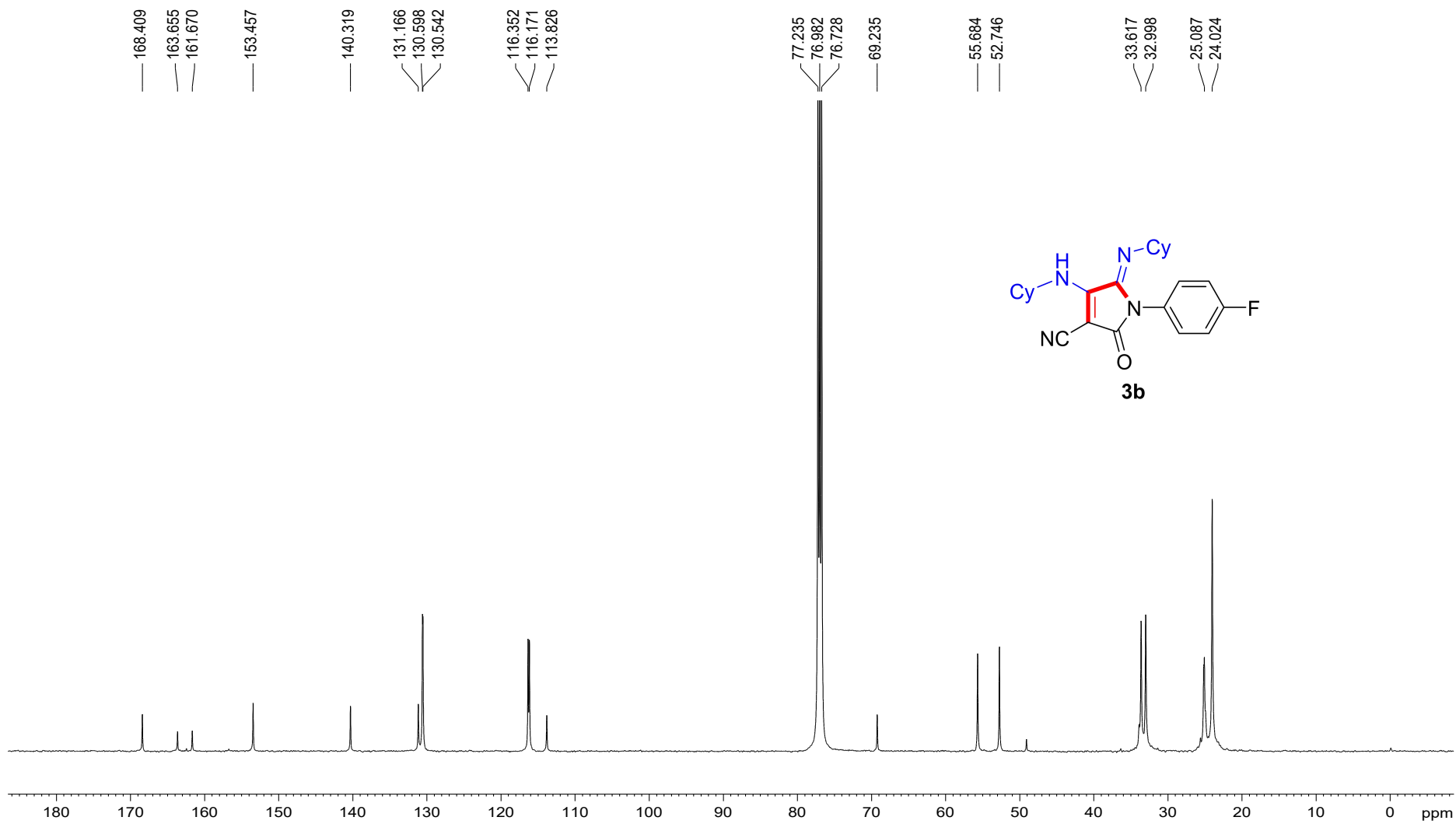
(s, 1H, CH), 3.13 (s, 1H, CH), 2.06 (s, 2H, CyH), 1.79 (s, 2H, CyH), 1.57 (s, 6H, CyH), 1.49 (s, 4H, CyH), 1.25 (s, 3H, CyH), 1.14 (s, 1H, CyH), 0.81 (s, 2H, CyH); ^{13}C NMR (CDCl_3 , 125 MHz) δ 171.11, 164.19, 156.81, 141.18, 138.37, 136.32, 129.24, 128.85, 128.62, 123.32, 119.66, 89.78, 56.46, 52.30, 33.59, 25.33, 24.64, 23.78; **HRMS** (ESI) m/z calcd for $\text{C}_{29}\text{H}_{35}\text{N}_4\text{O}_2^+$ (M+H) $^+$ 471.2760, found 471.2756.

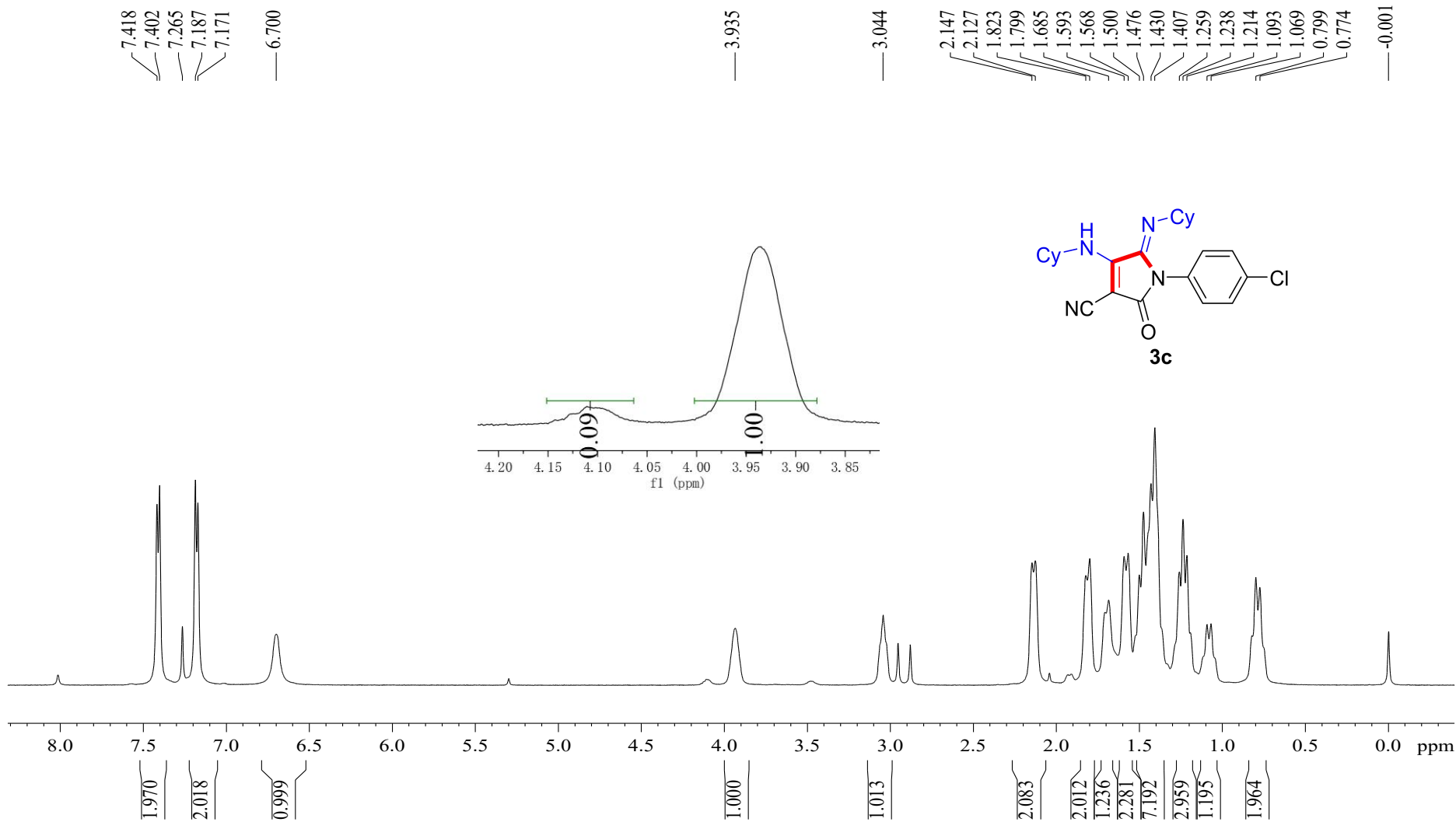




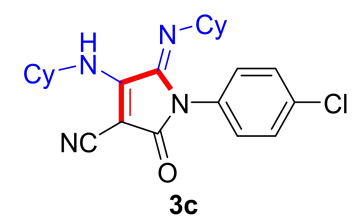
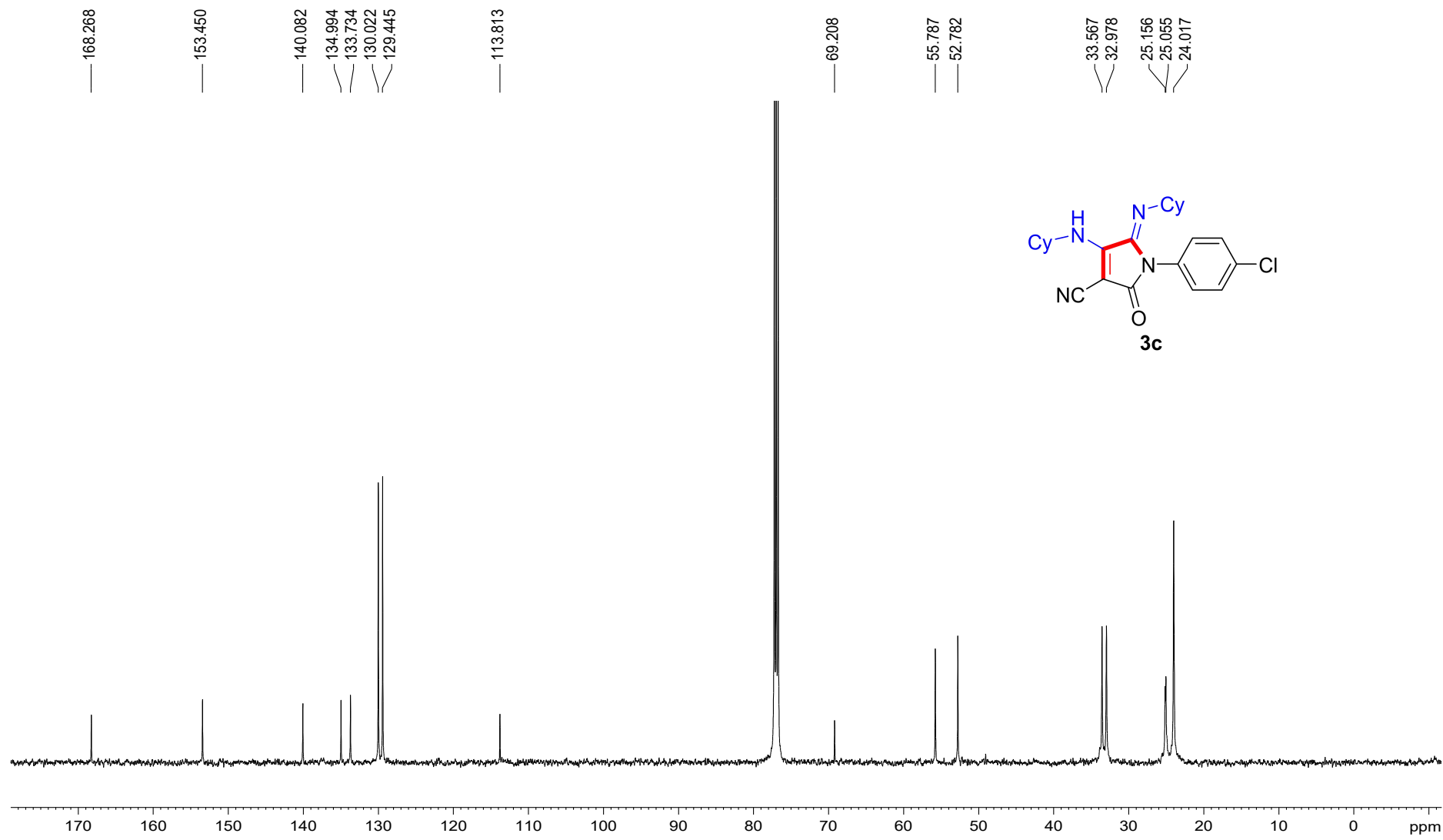
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7.207
7.144
7.127
7.110
3.019
3.012
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2.150
2.131
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1.835
1.827
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1.686
1.679
1.596
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1.225
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1.075
1.057
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0.761
0.754
-0.001

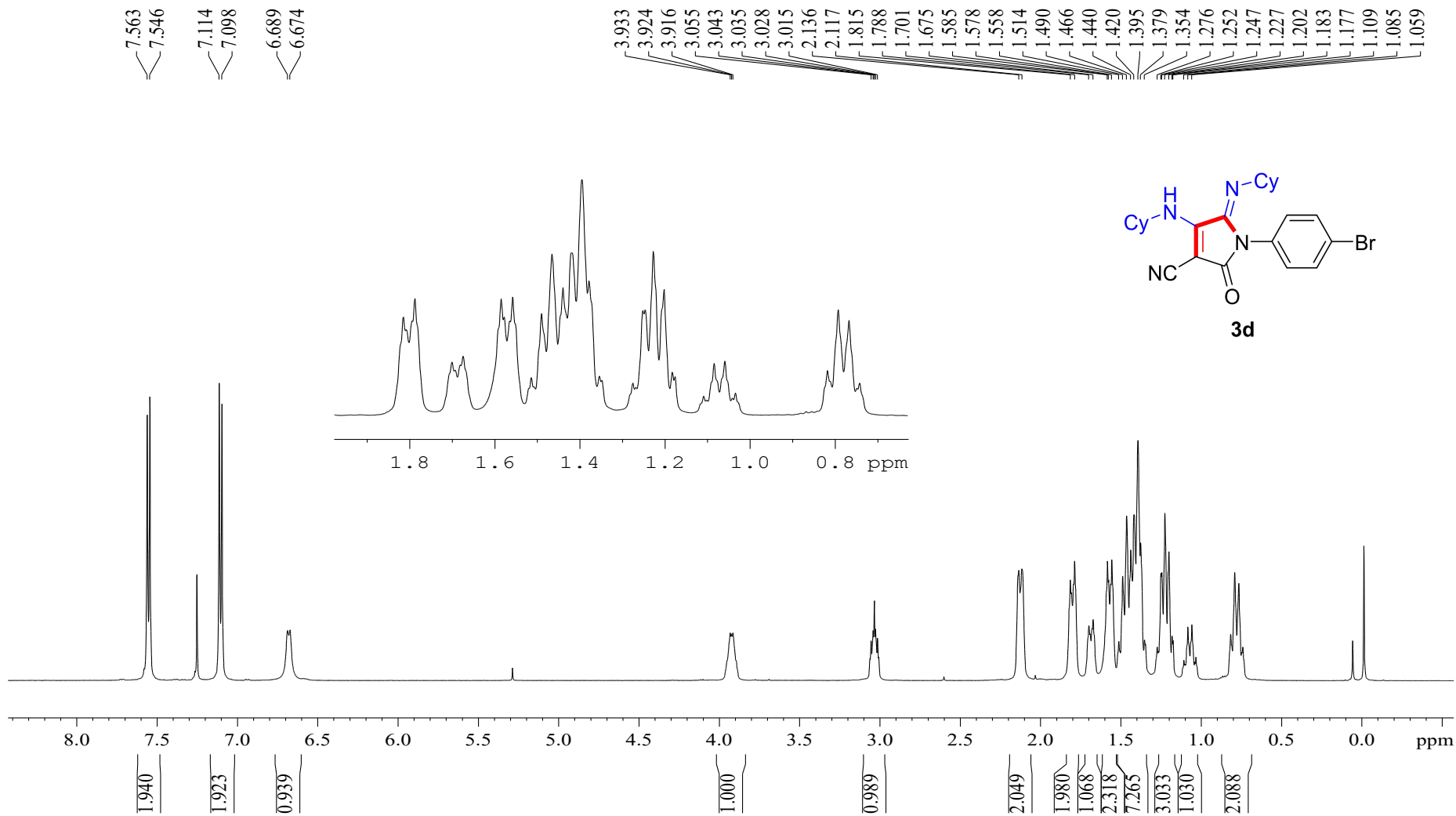




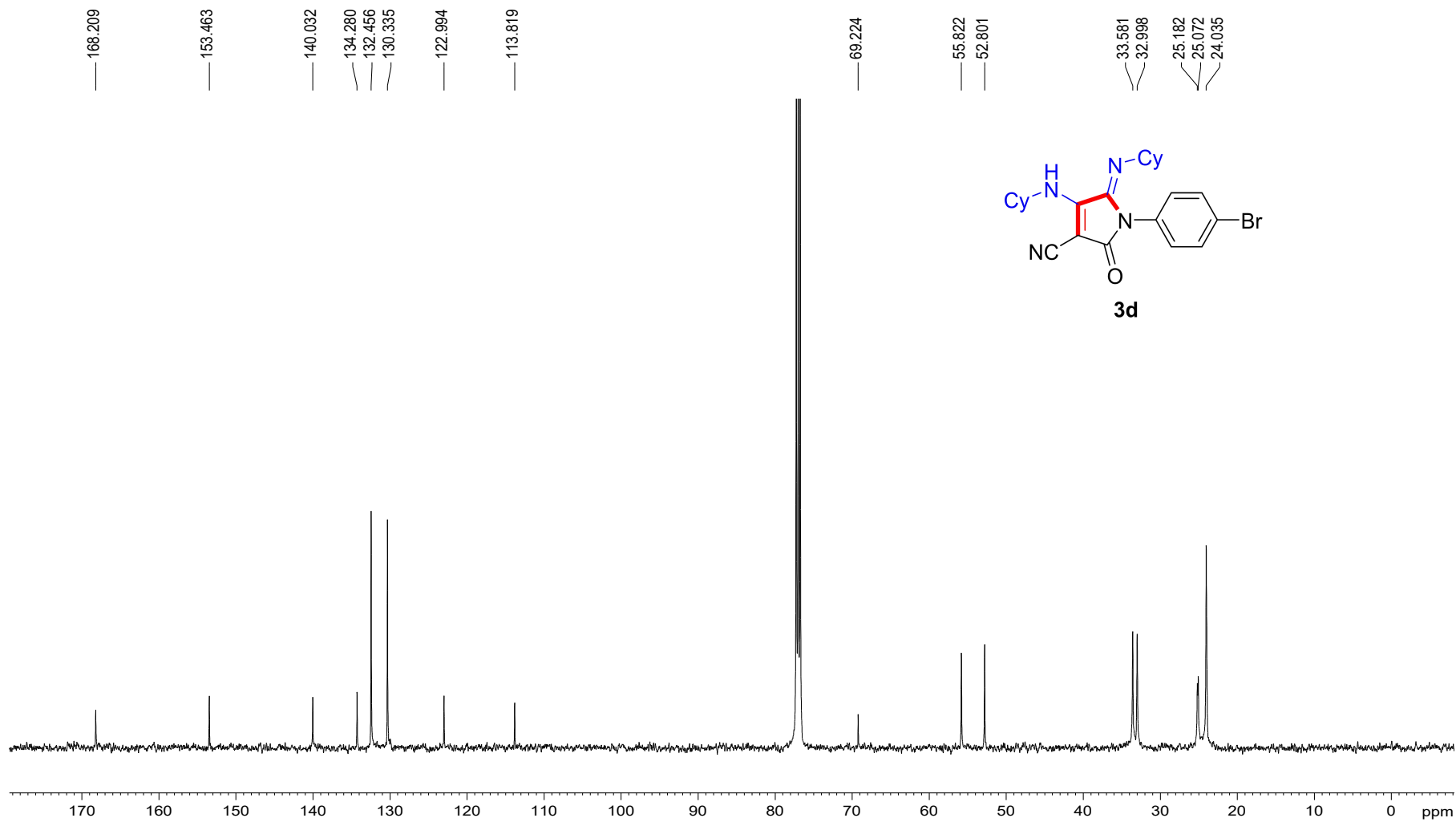


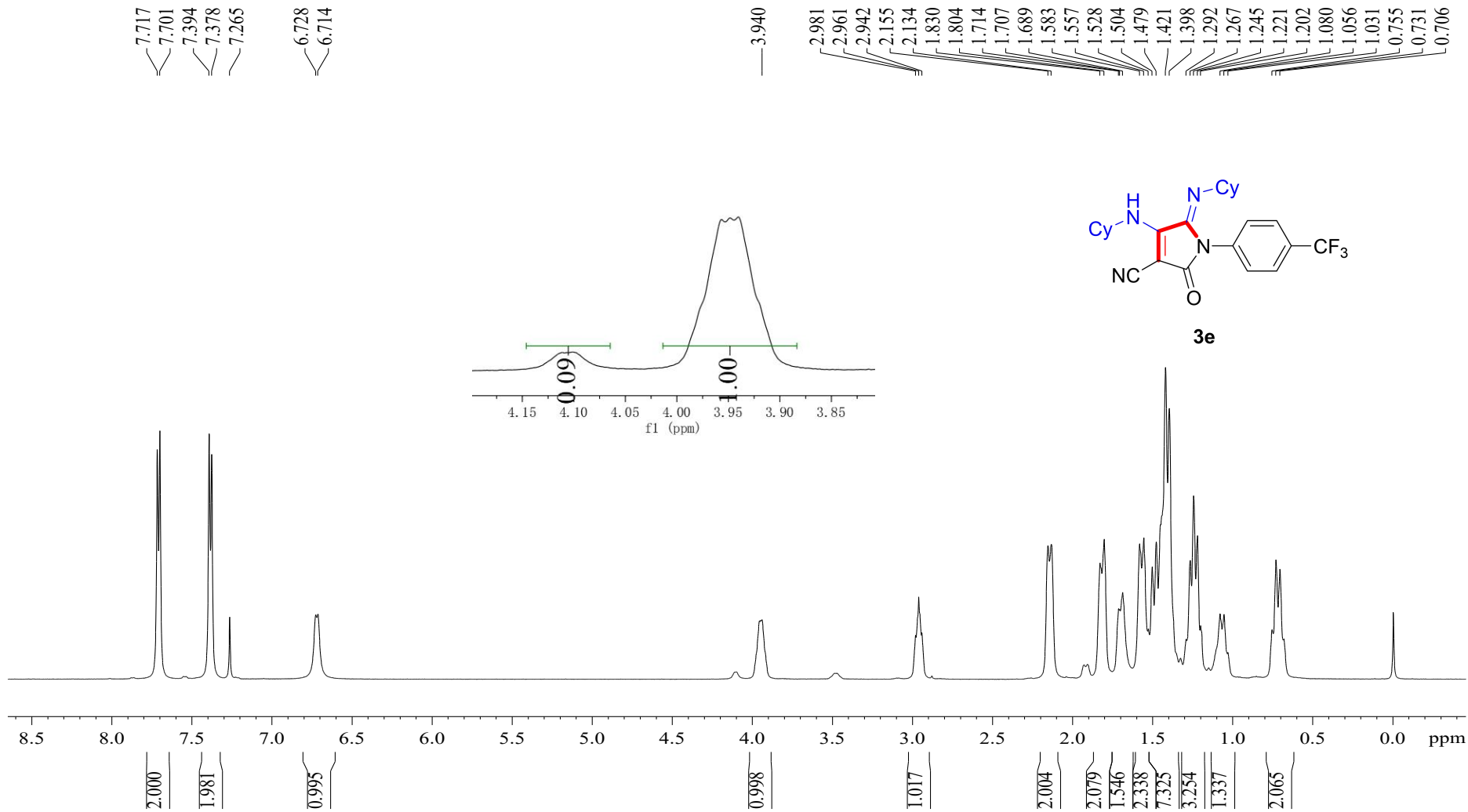
NJB-160812 13C 1D 2016 08 12

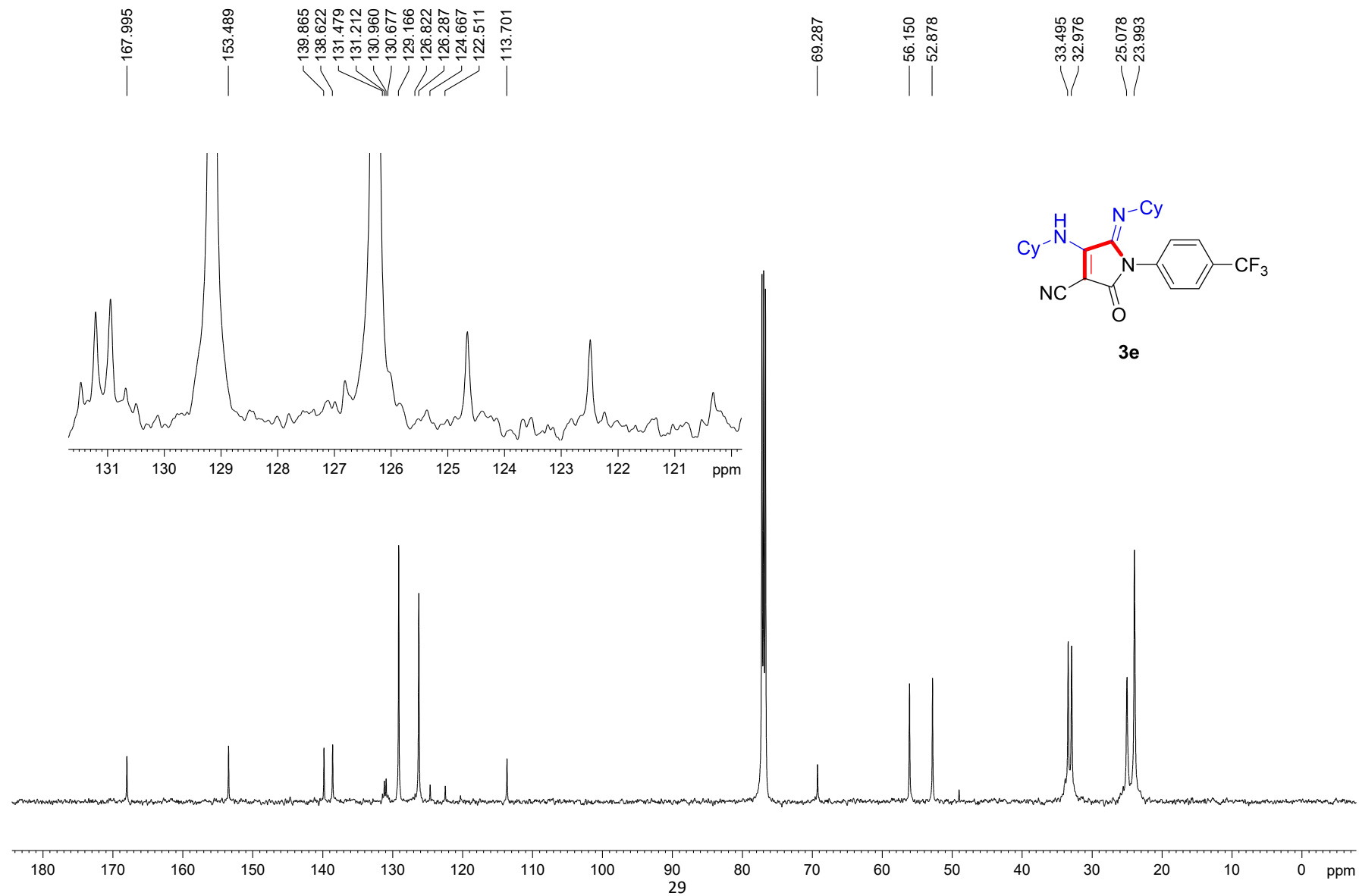


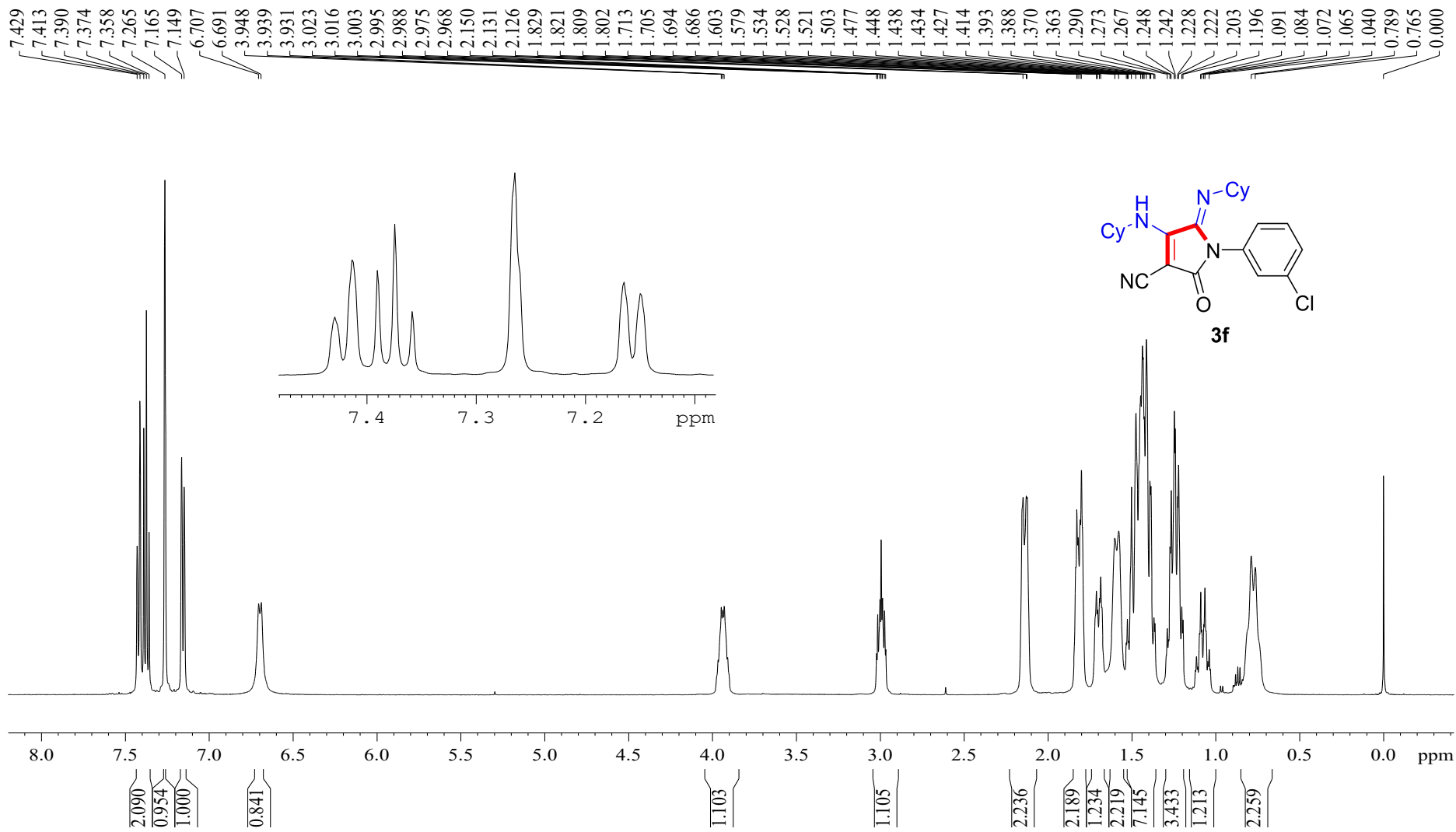


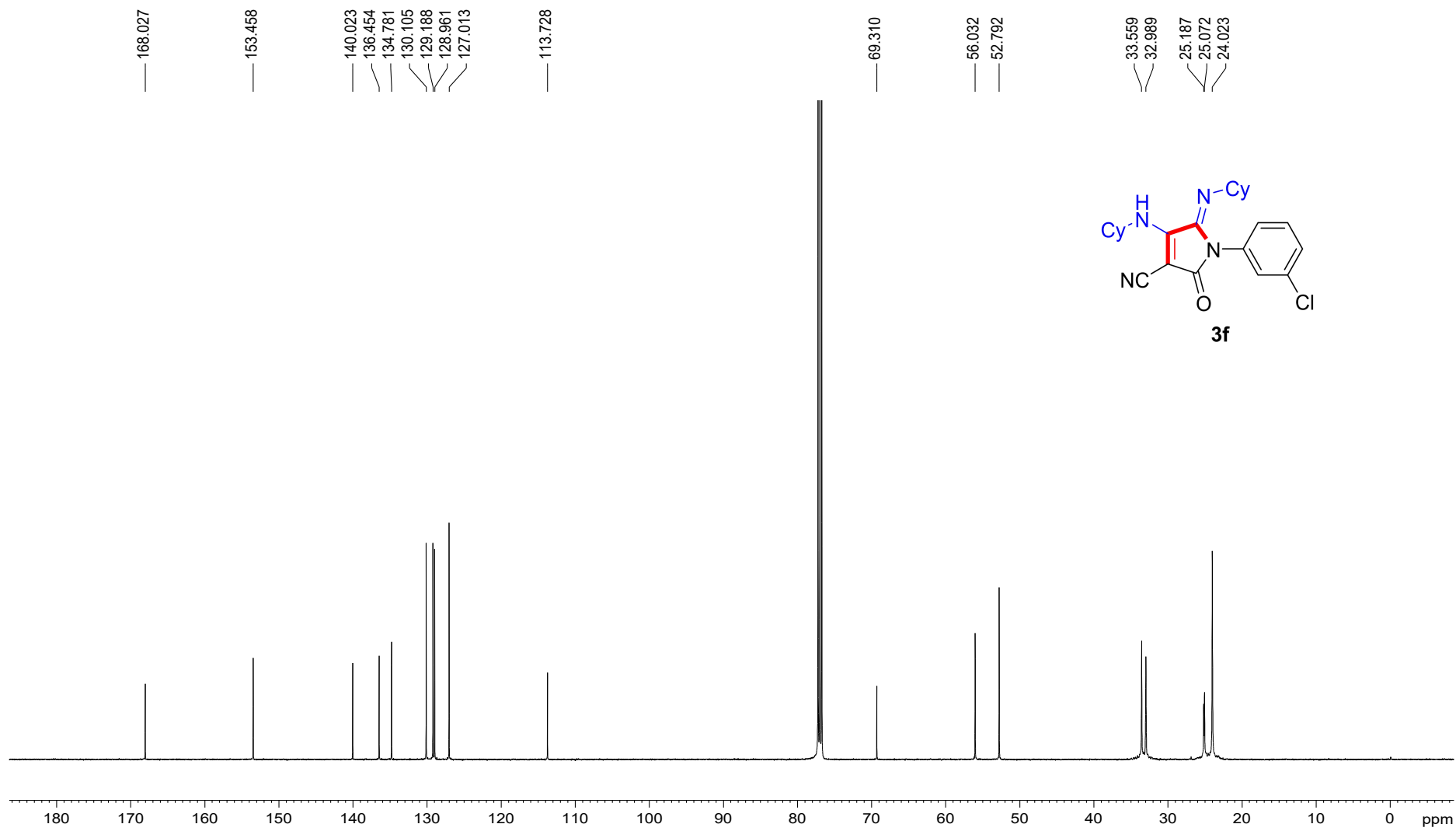
NJB-160523 13C 2016 05 25

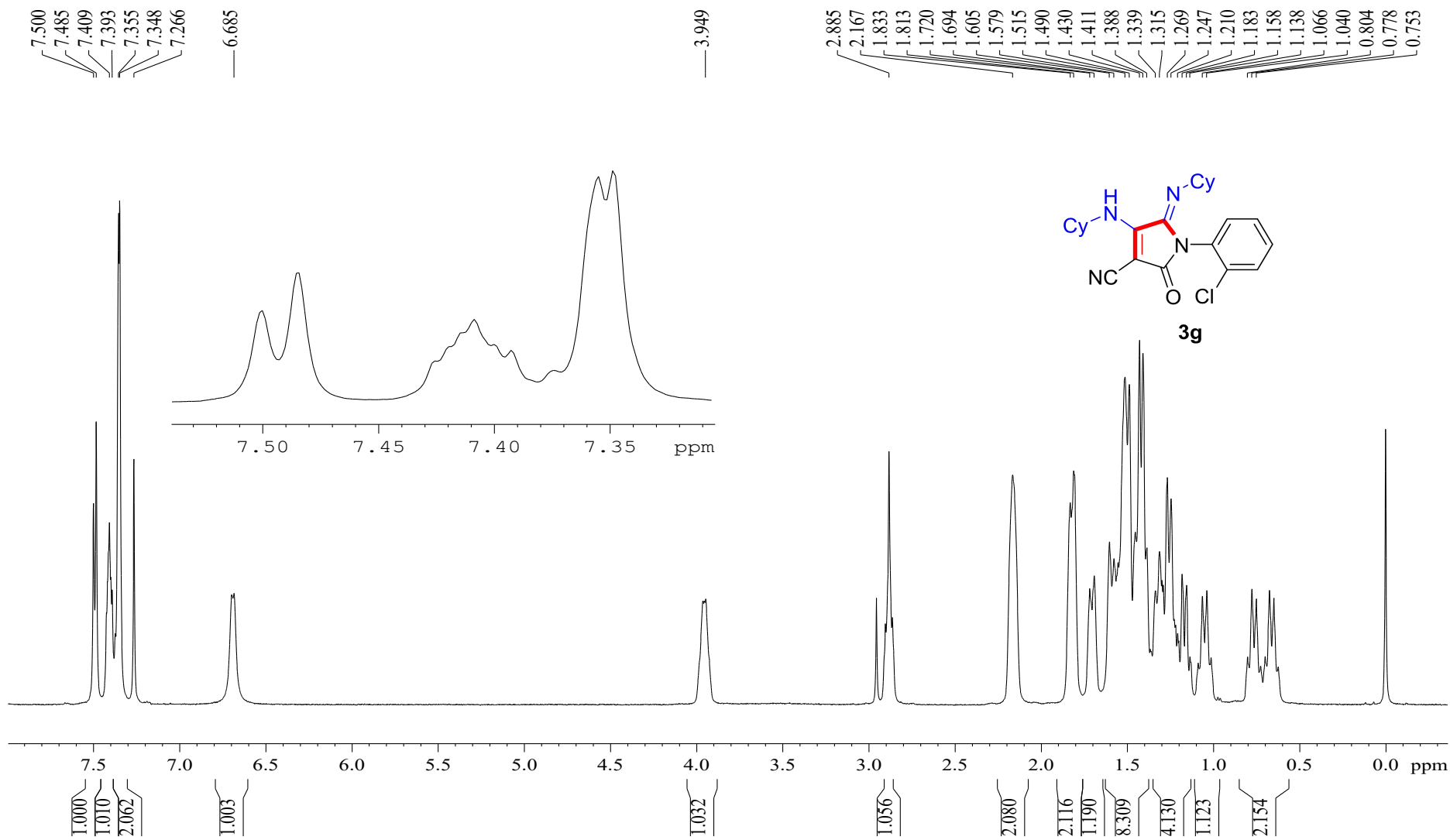


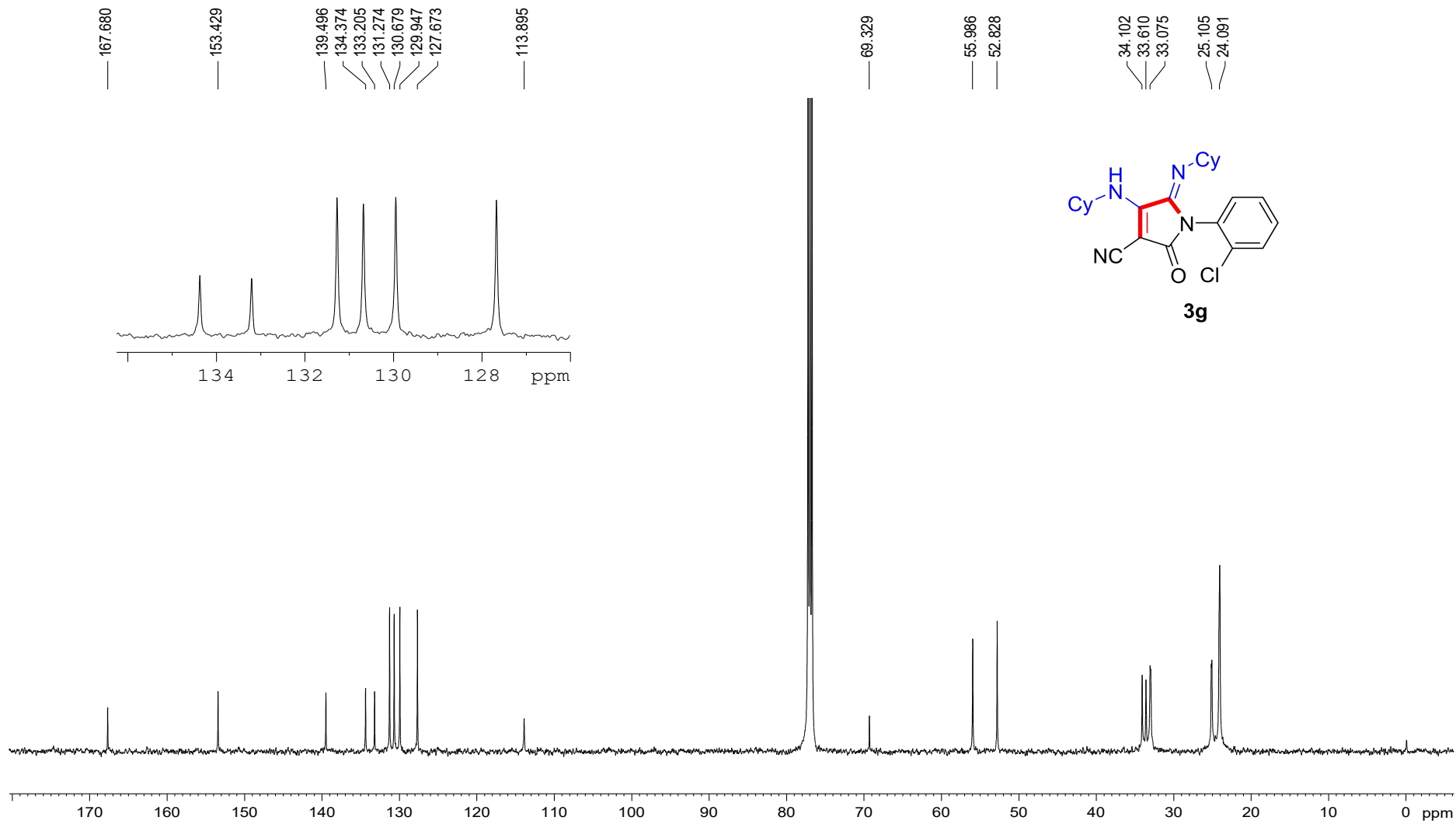


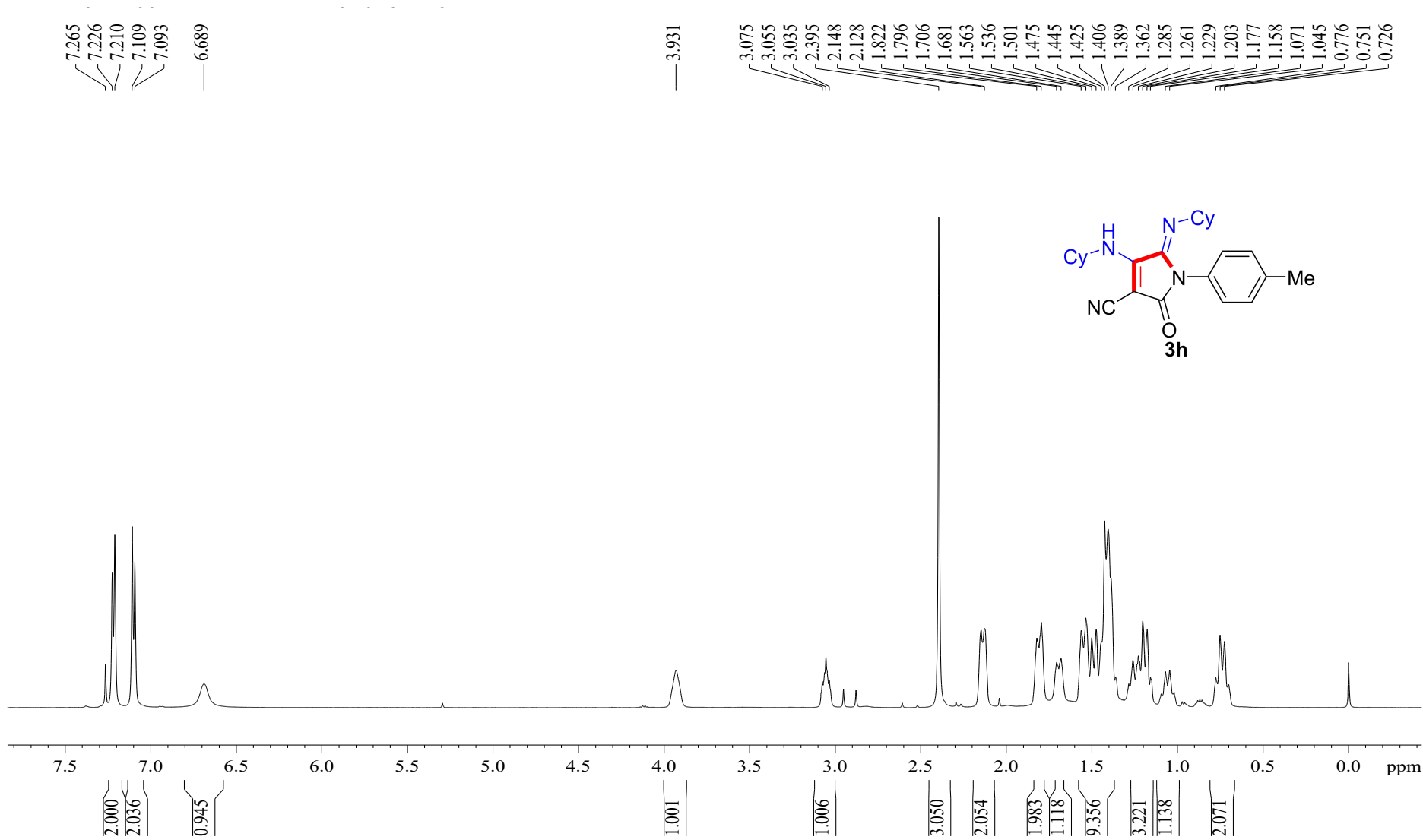




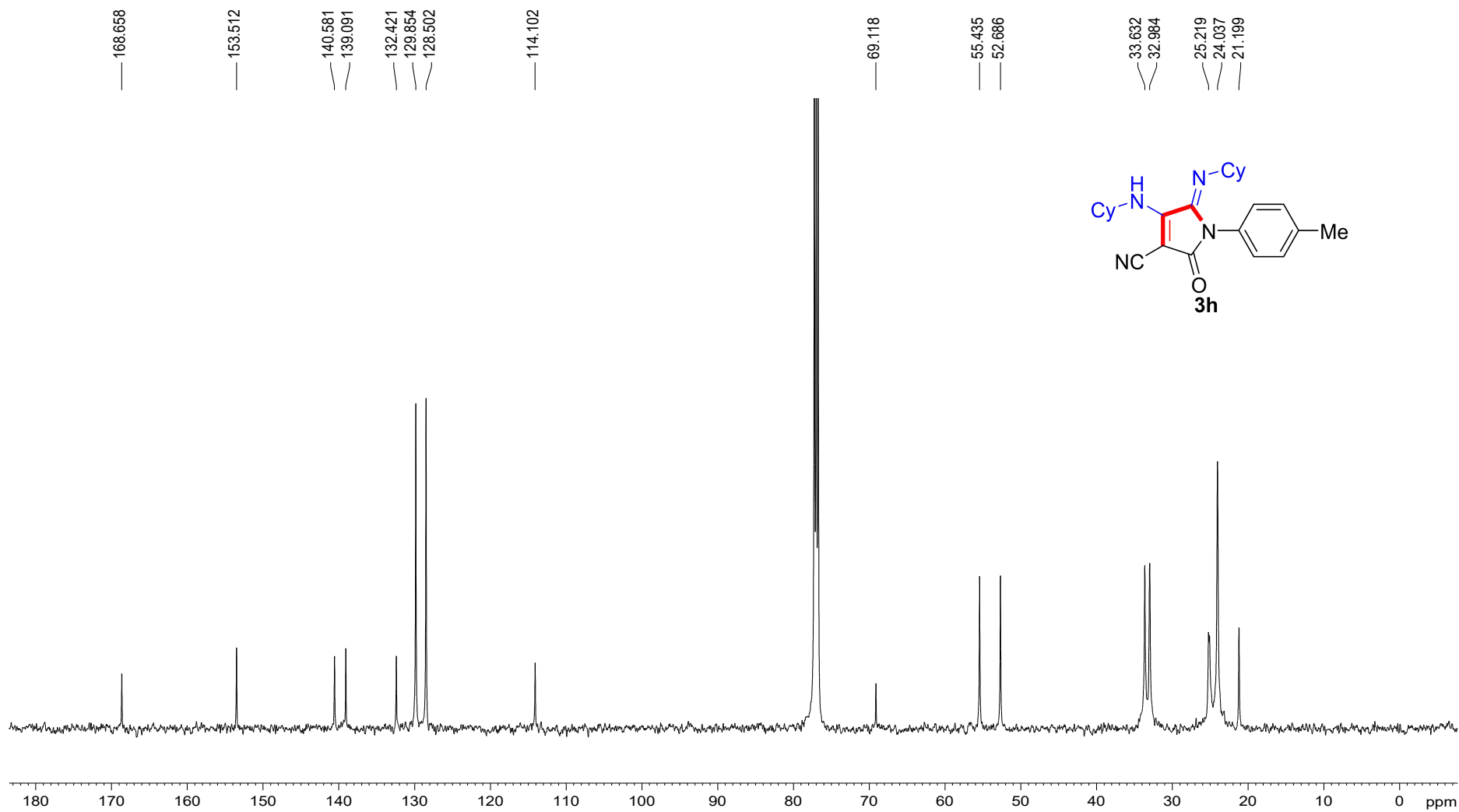


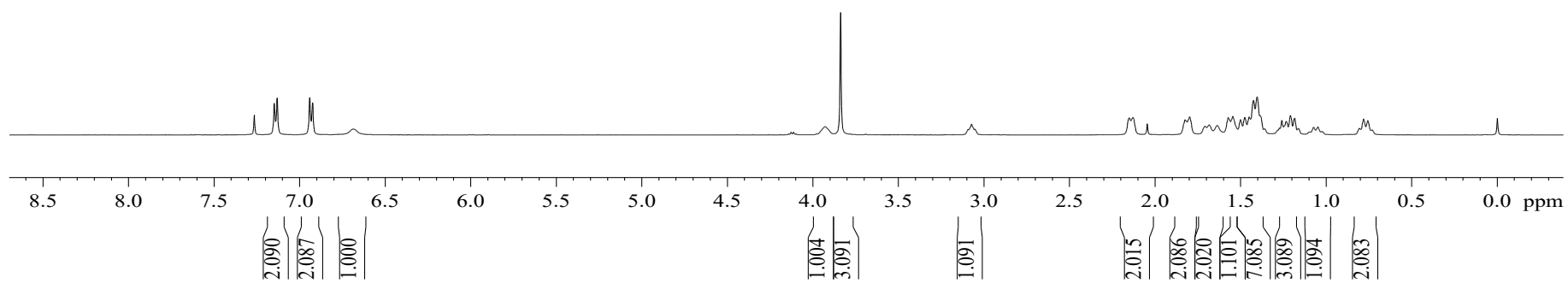
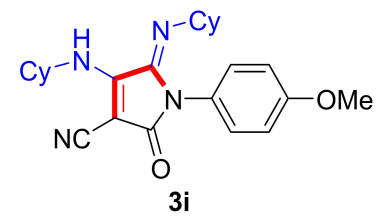
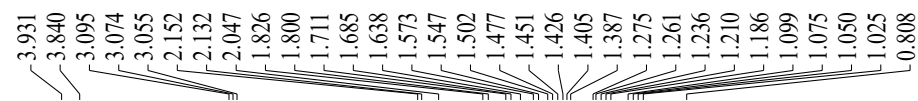
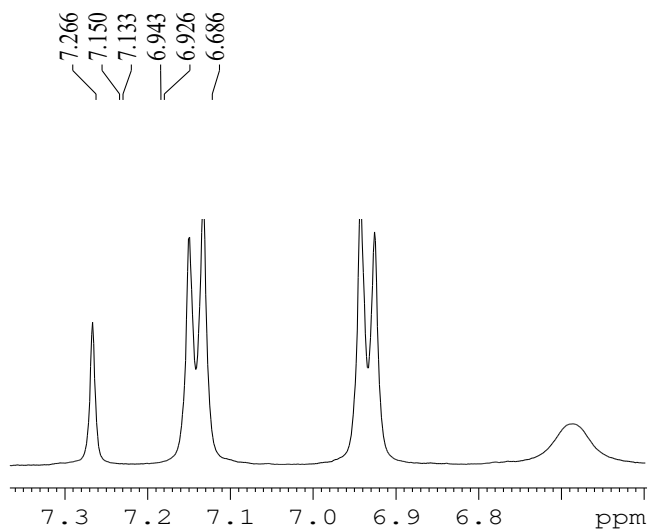


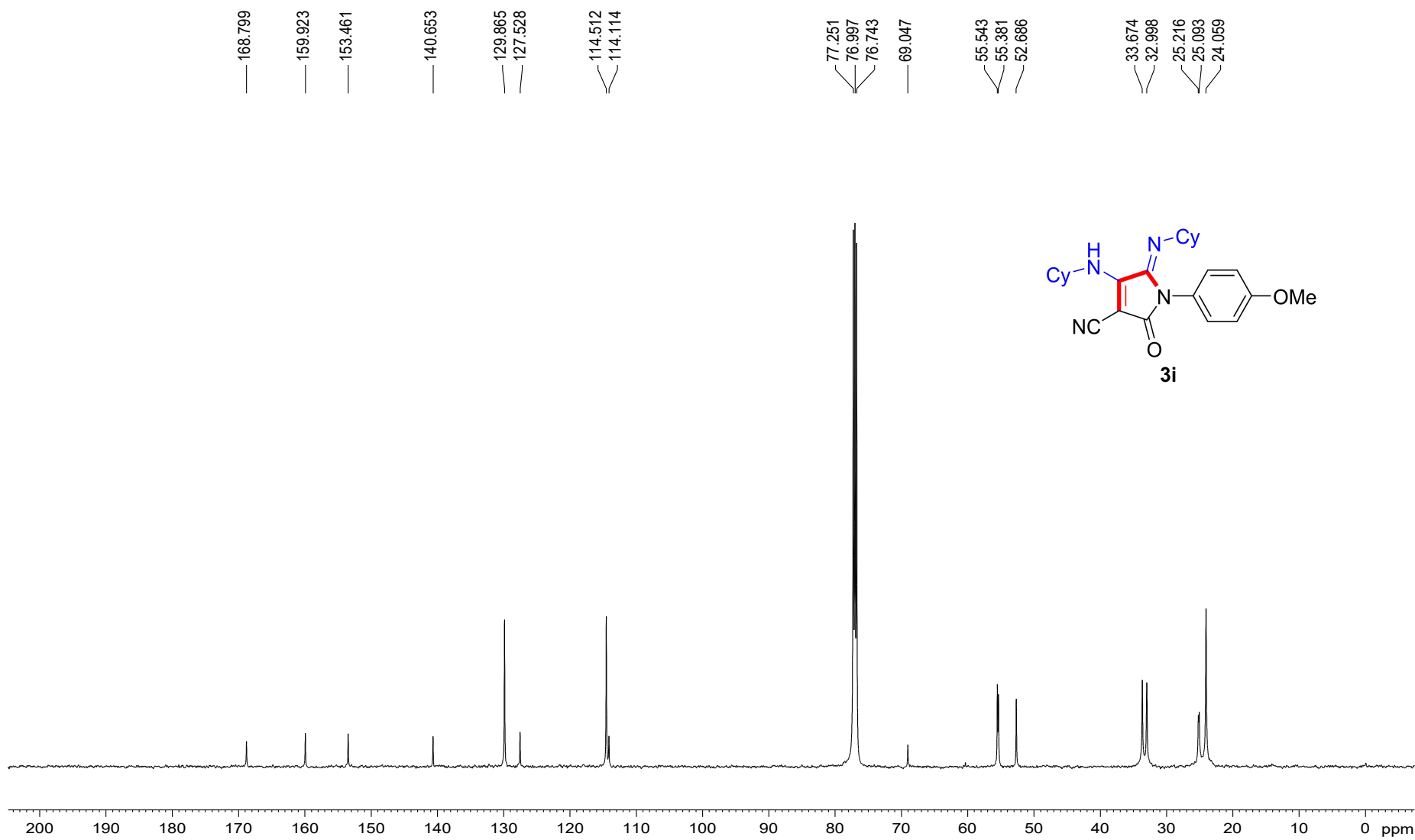


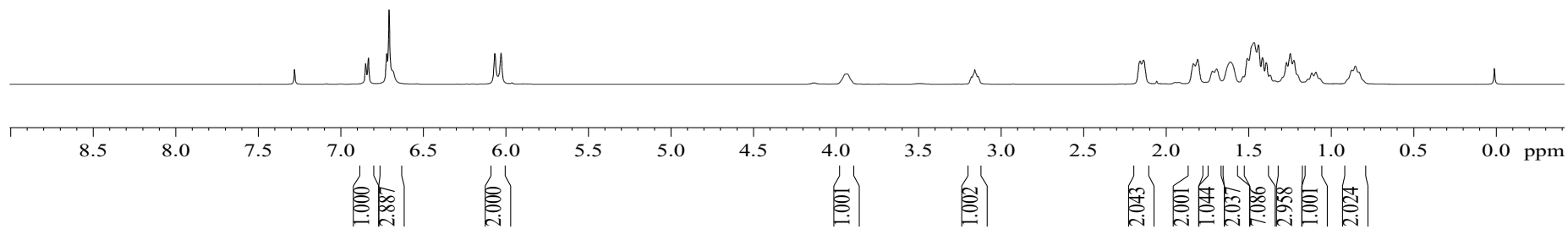
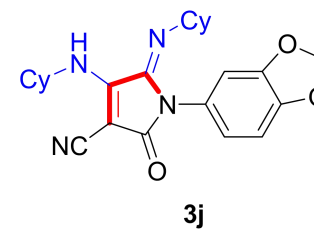
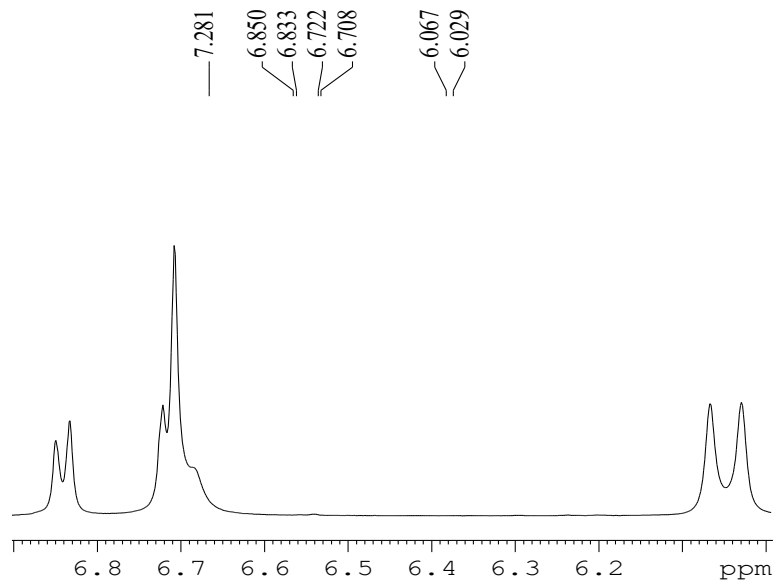


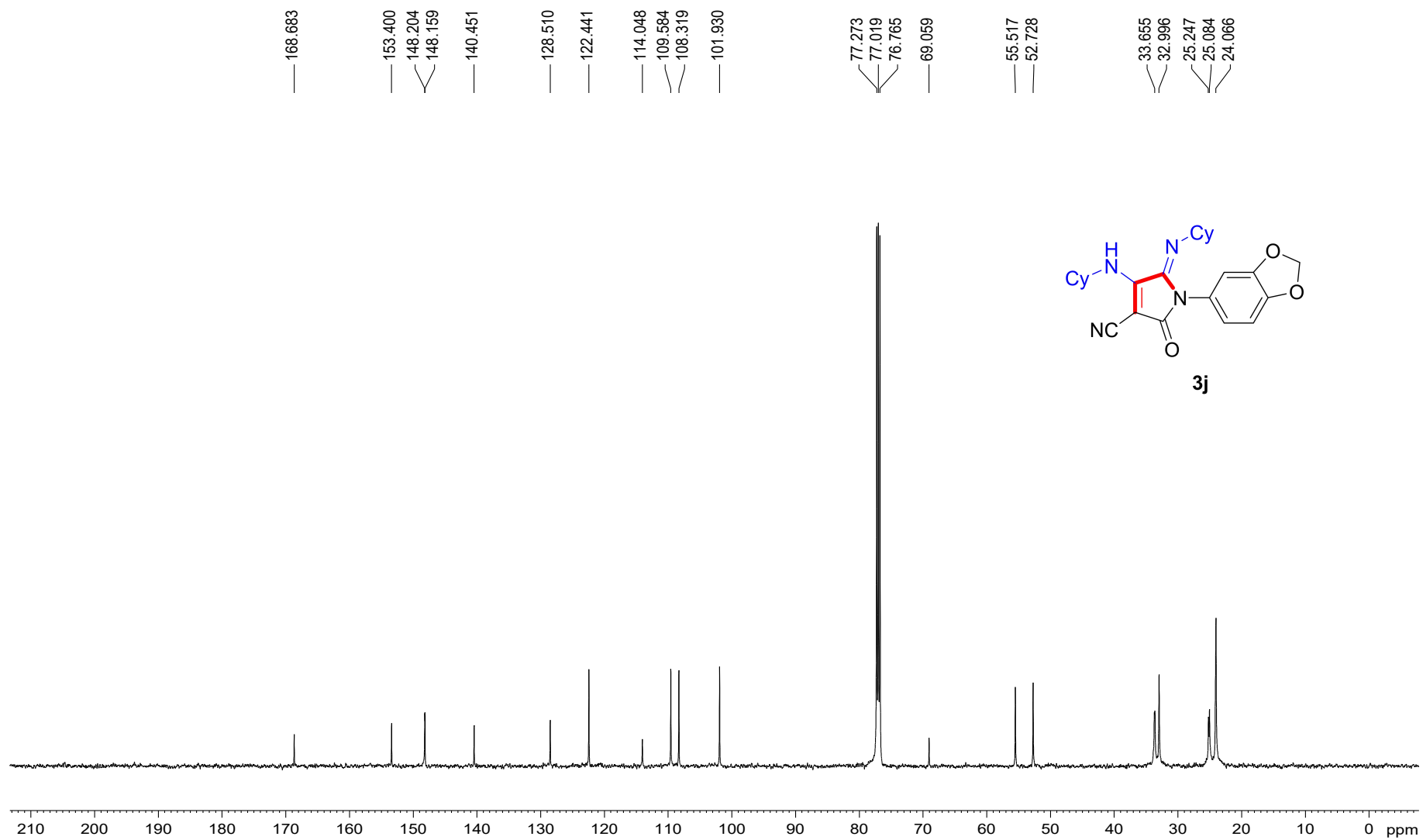
NJB-160727 13C 1D 2016 07 29

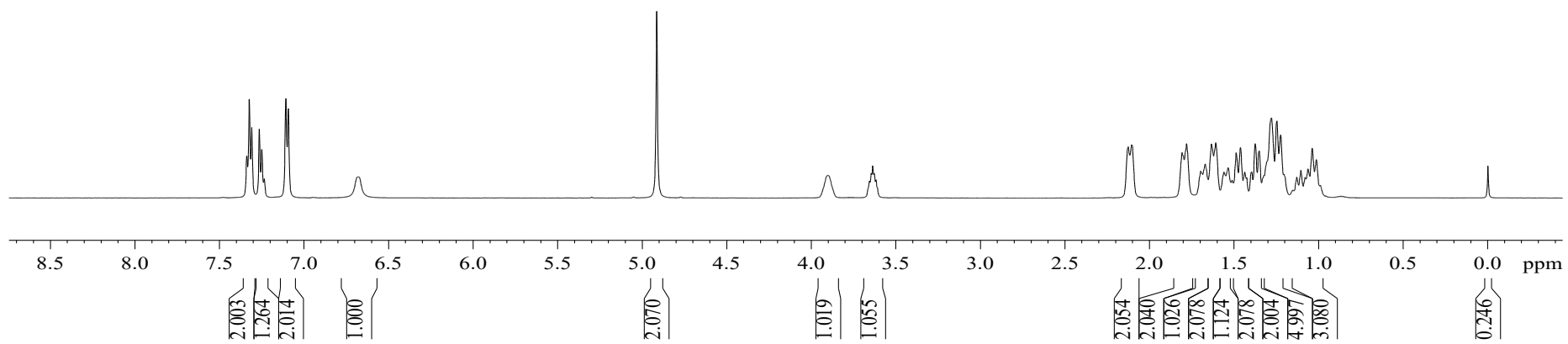
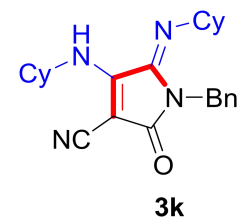
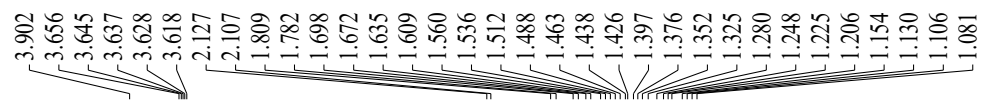
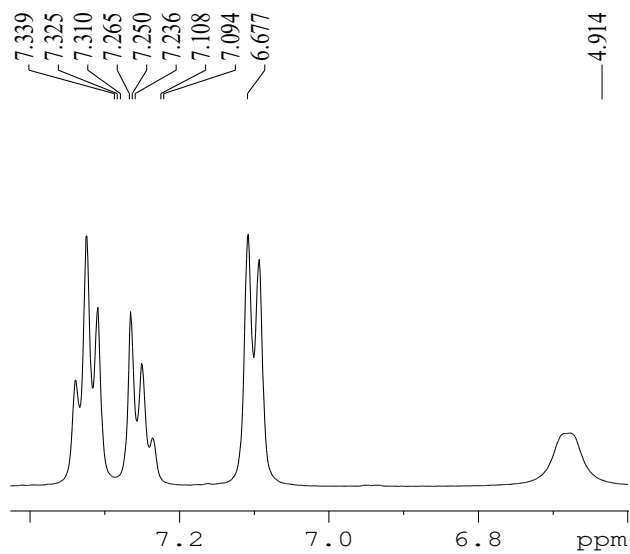


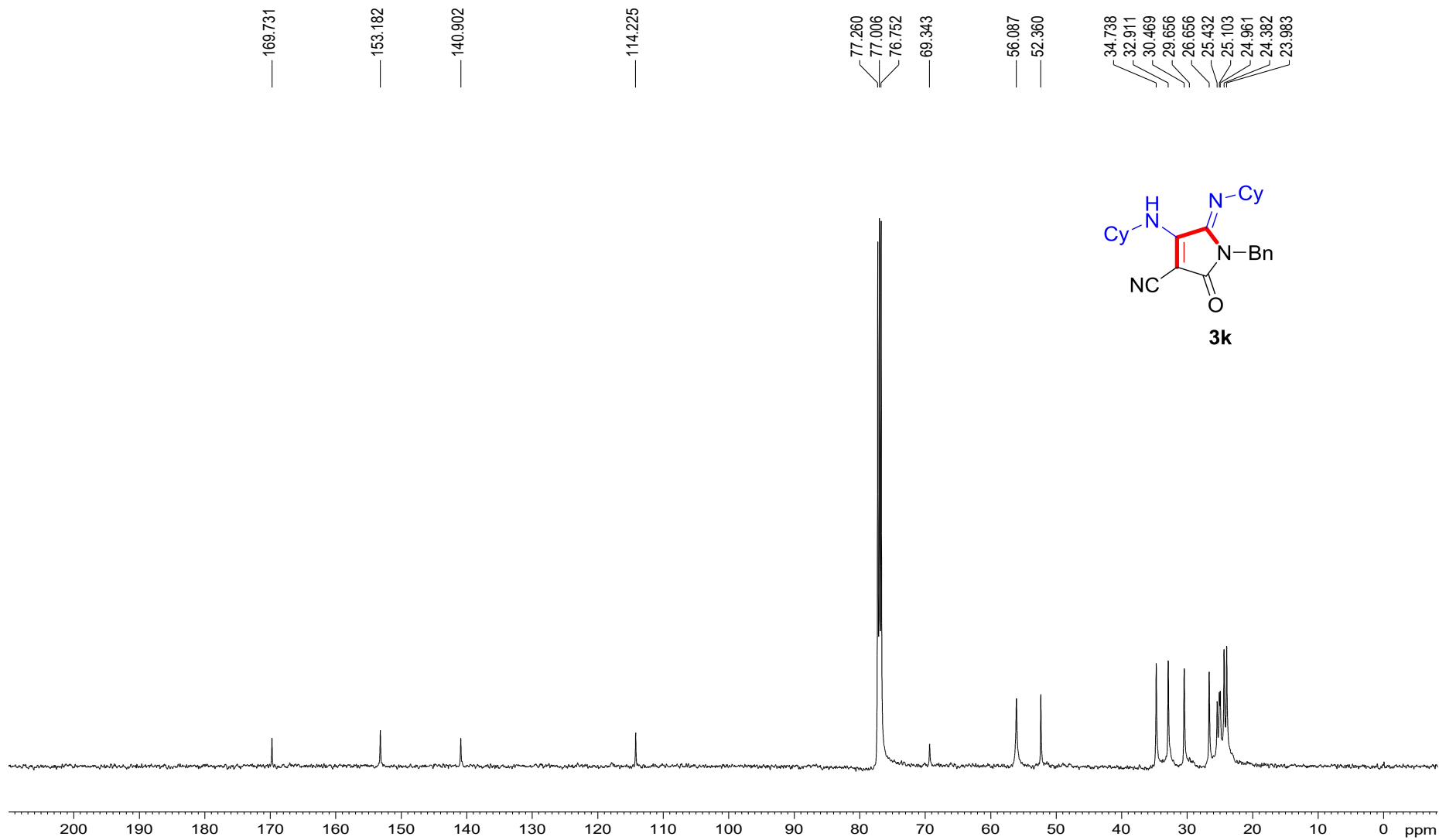








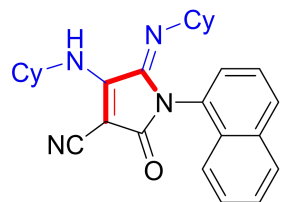




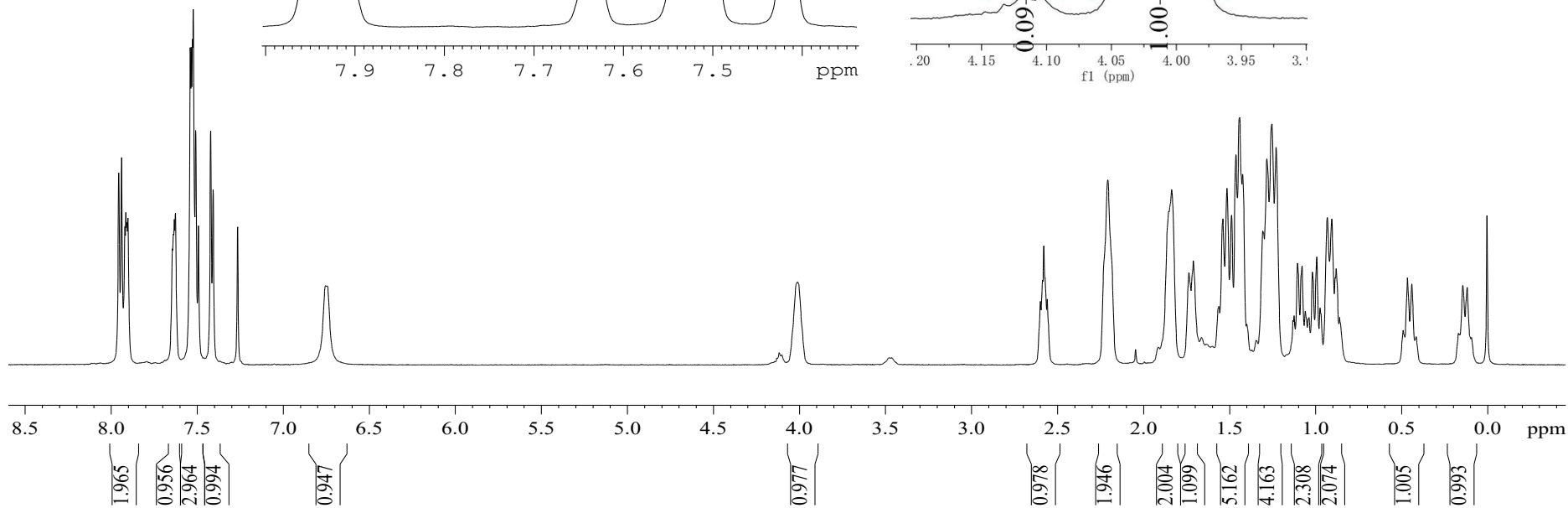
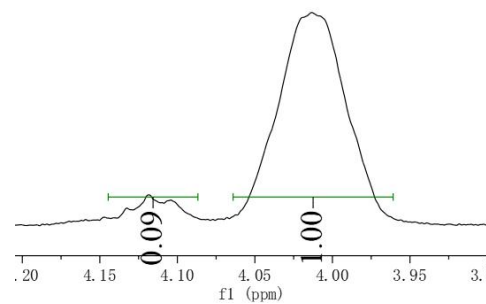
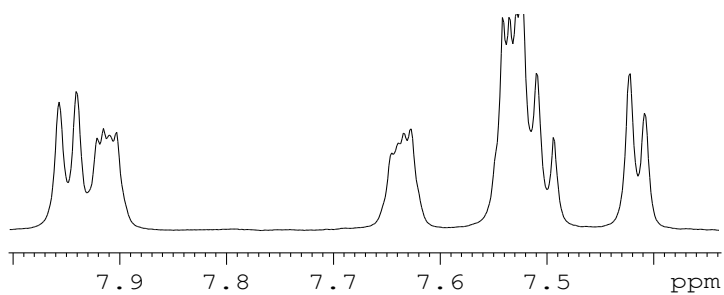
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6.755

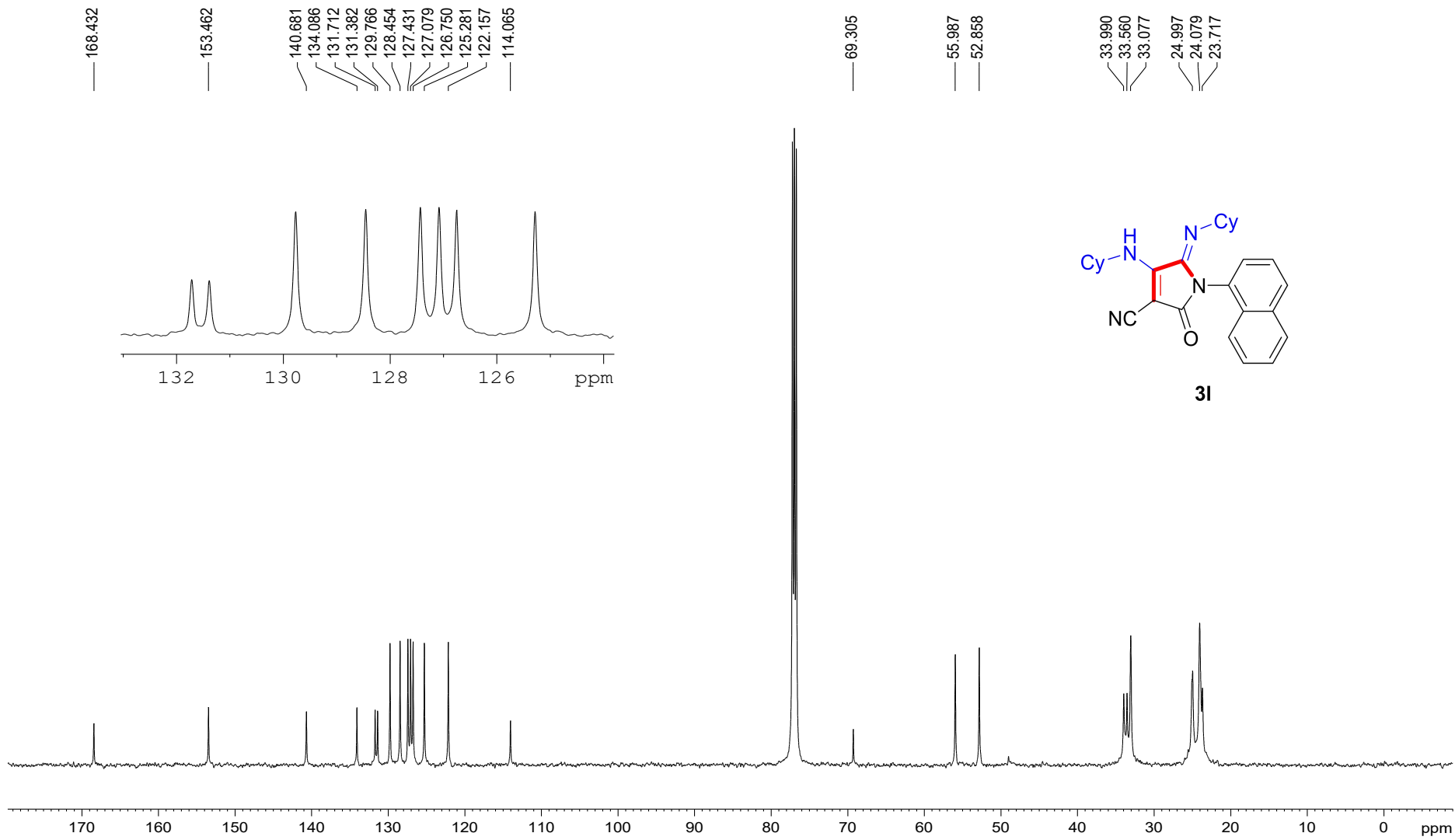
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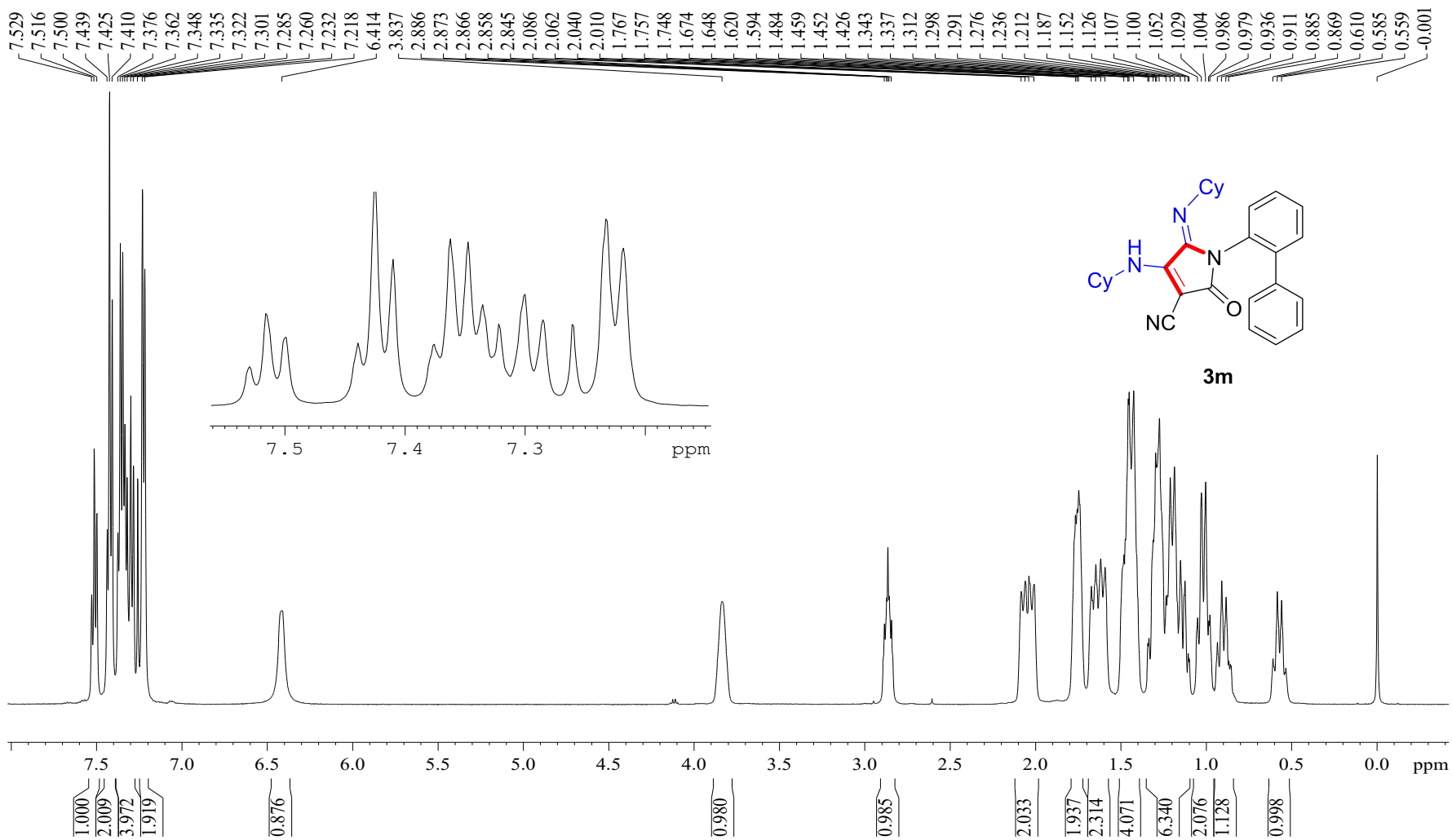
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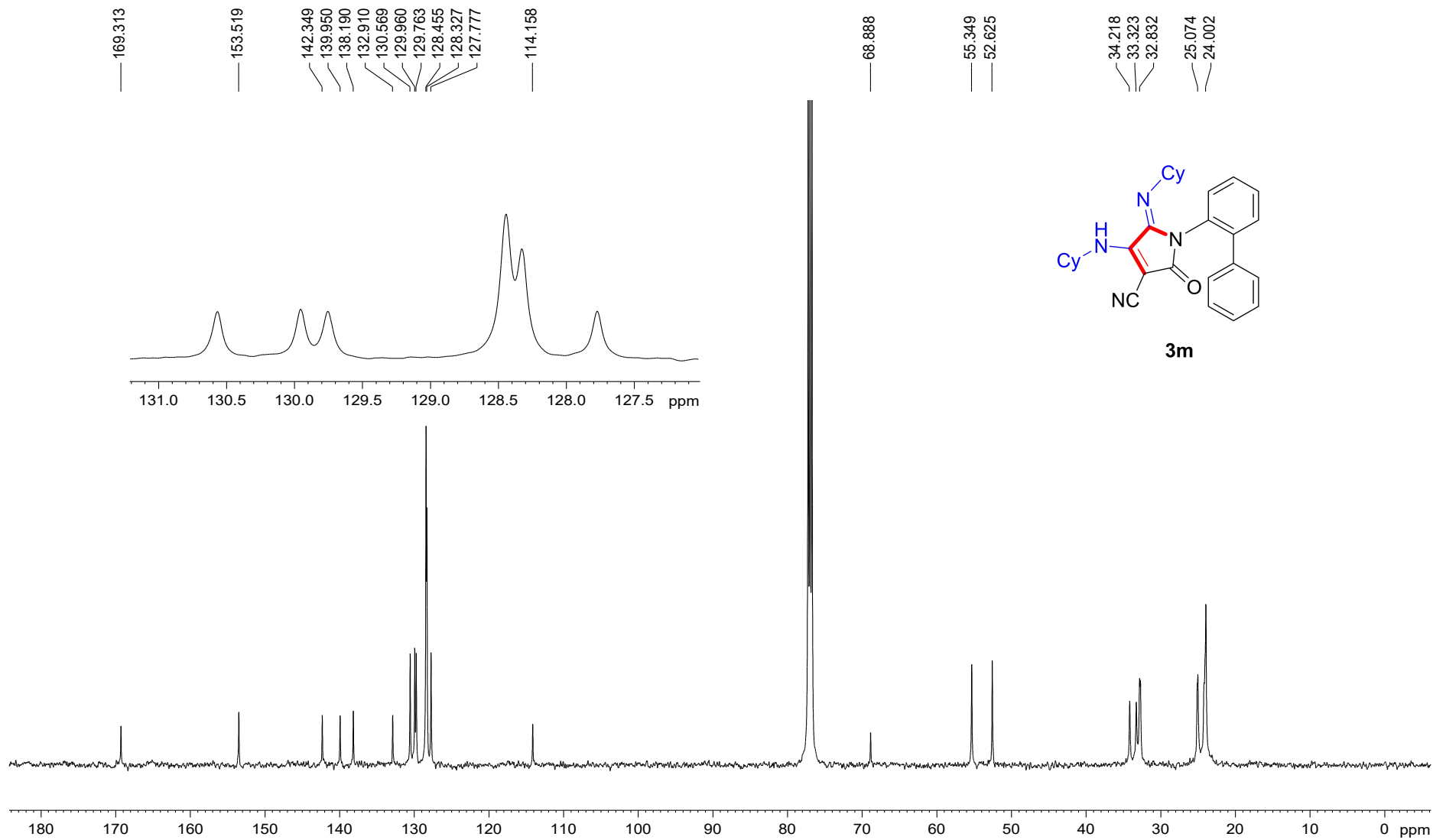


3I

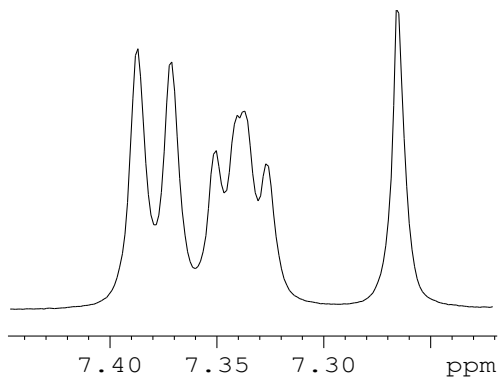




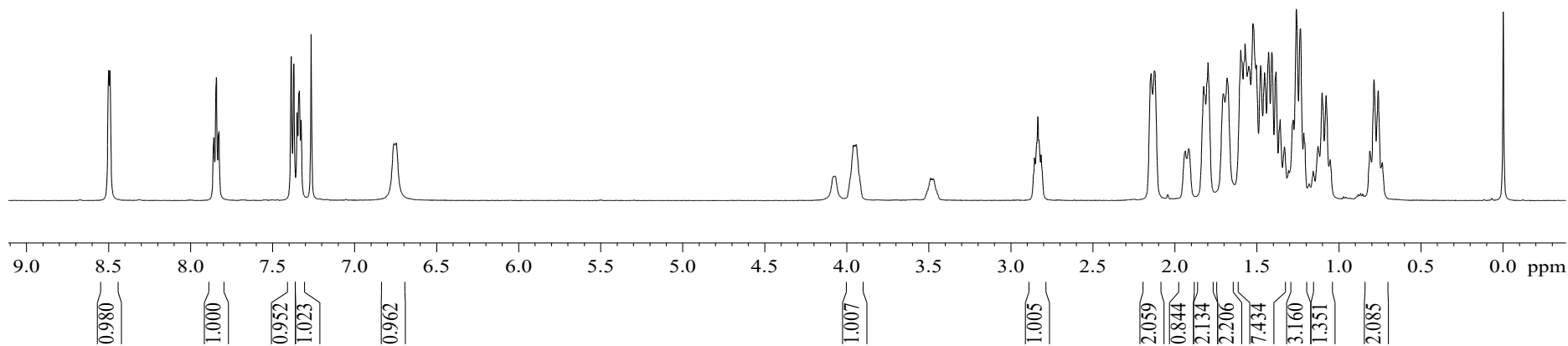
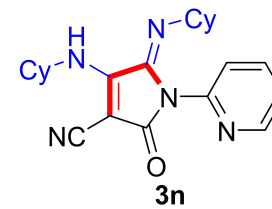
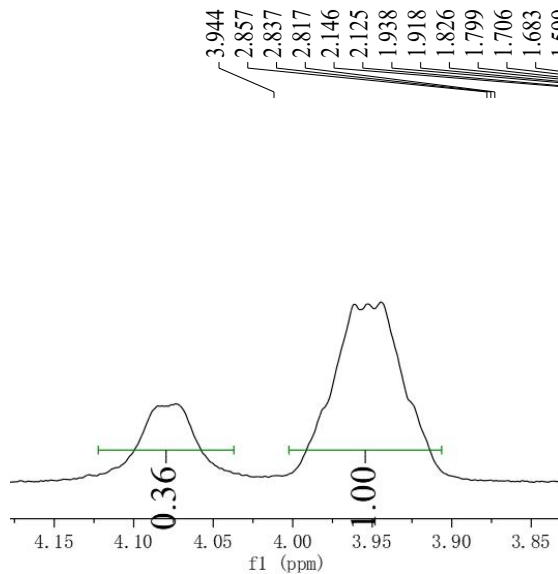


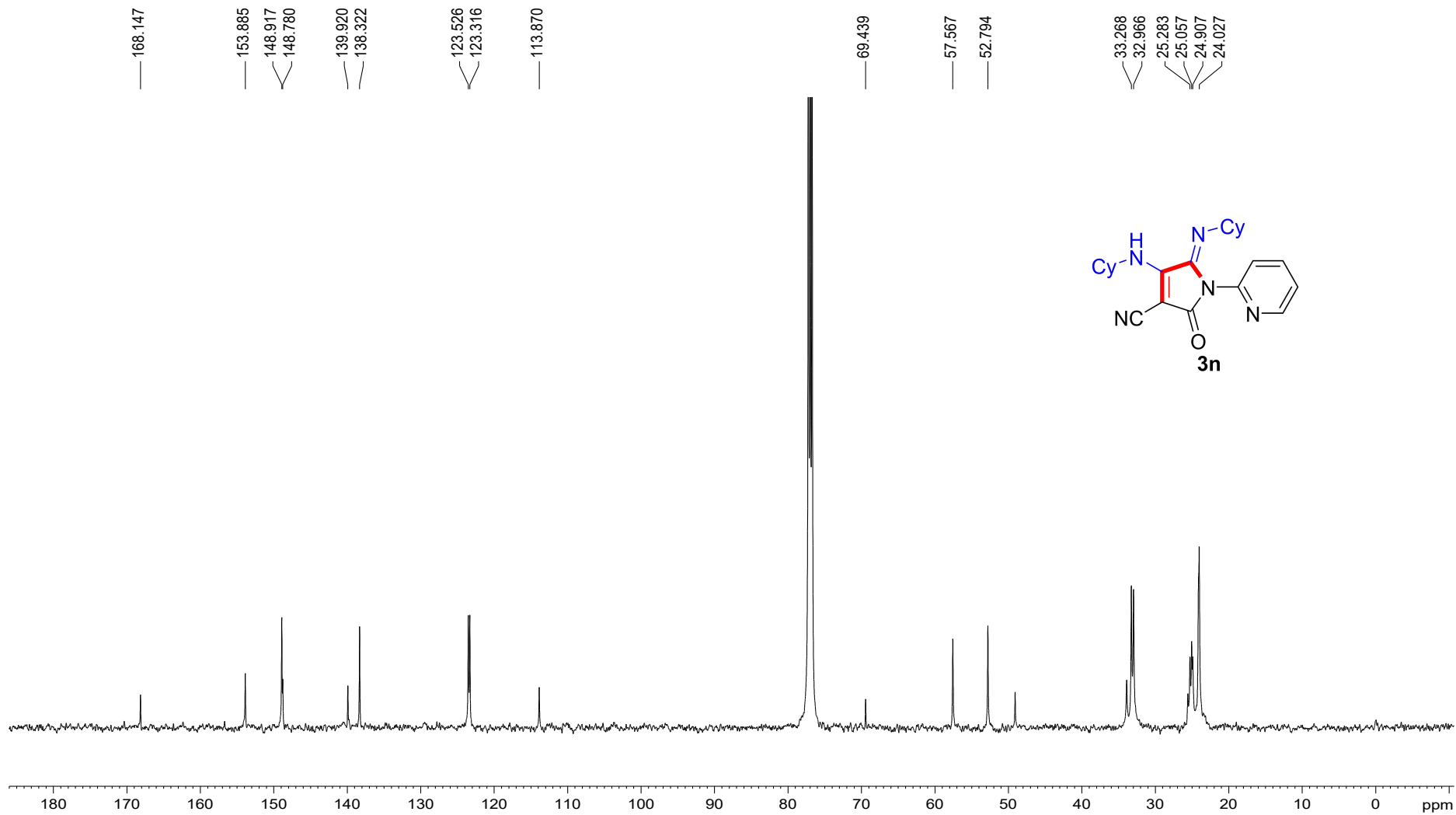


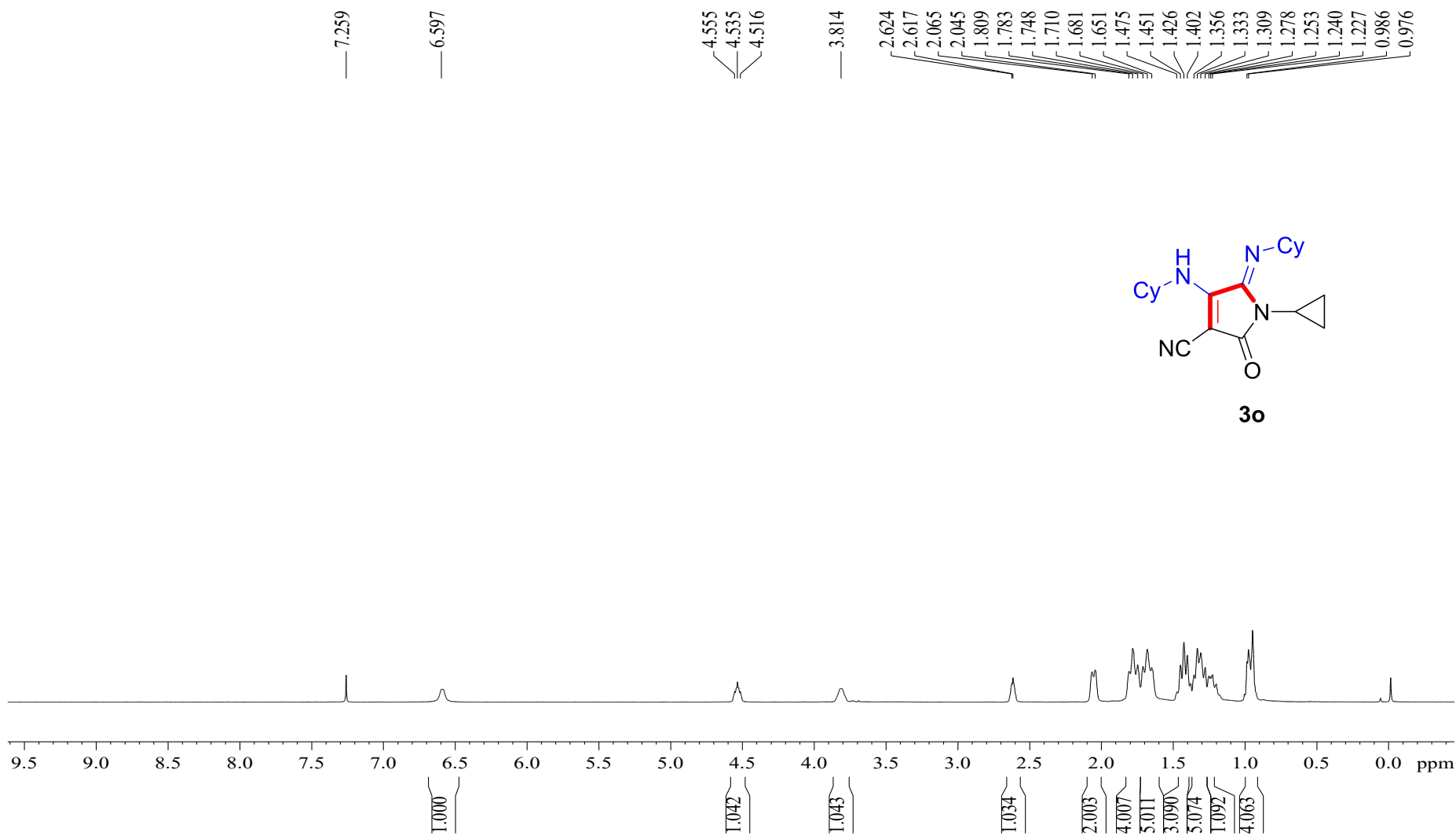
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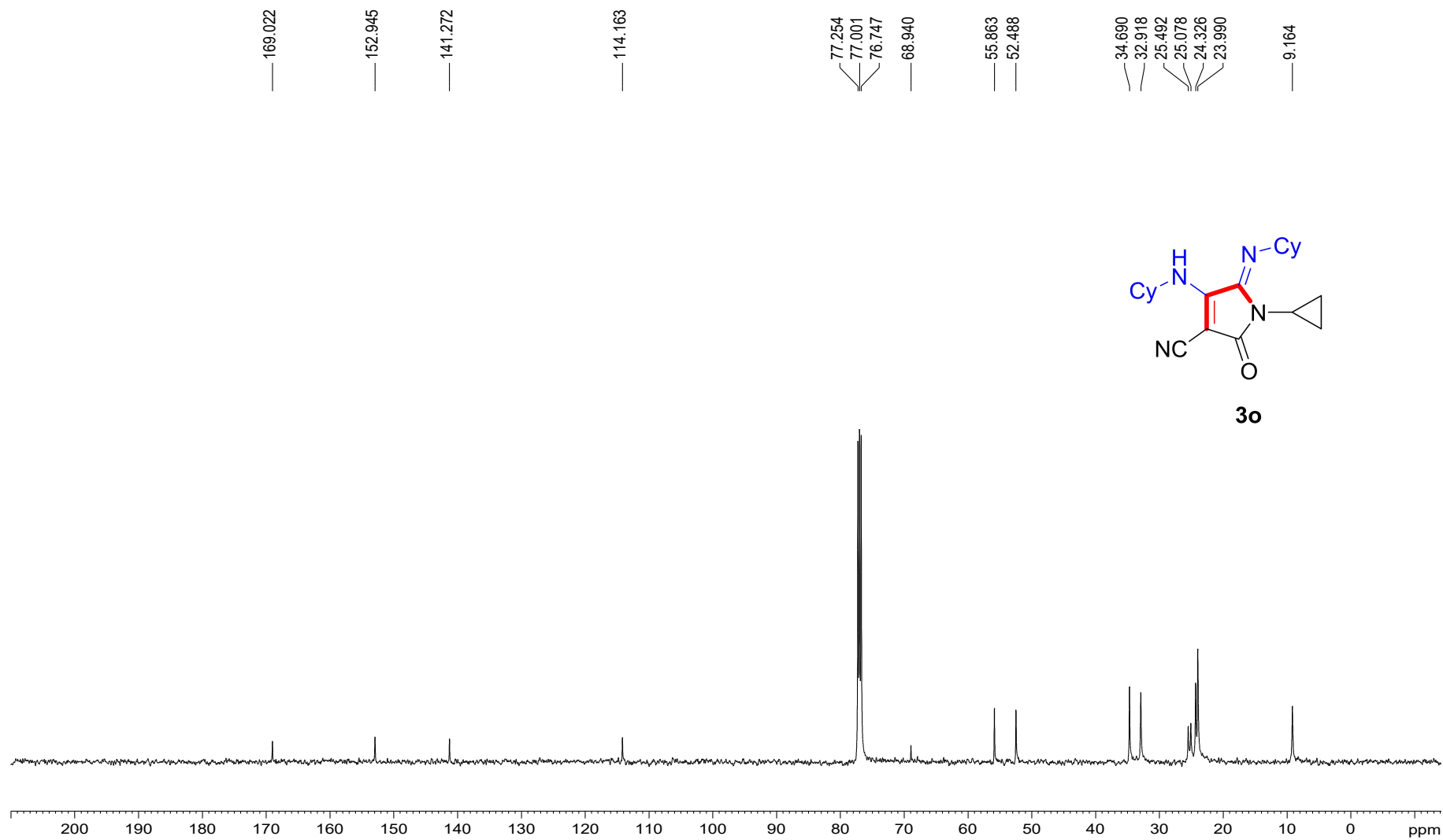


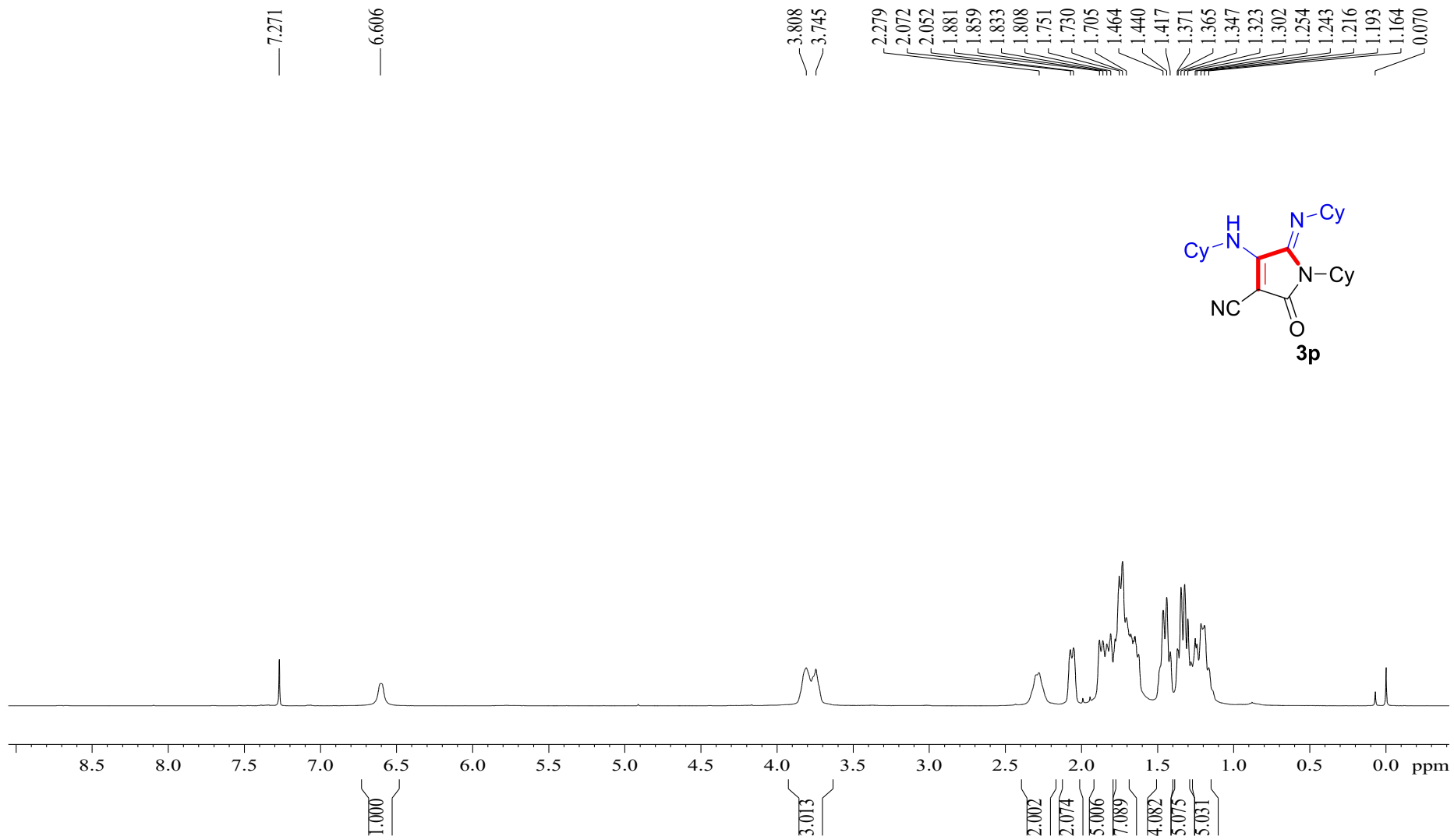
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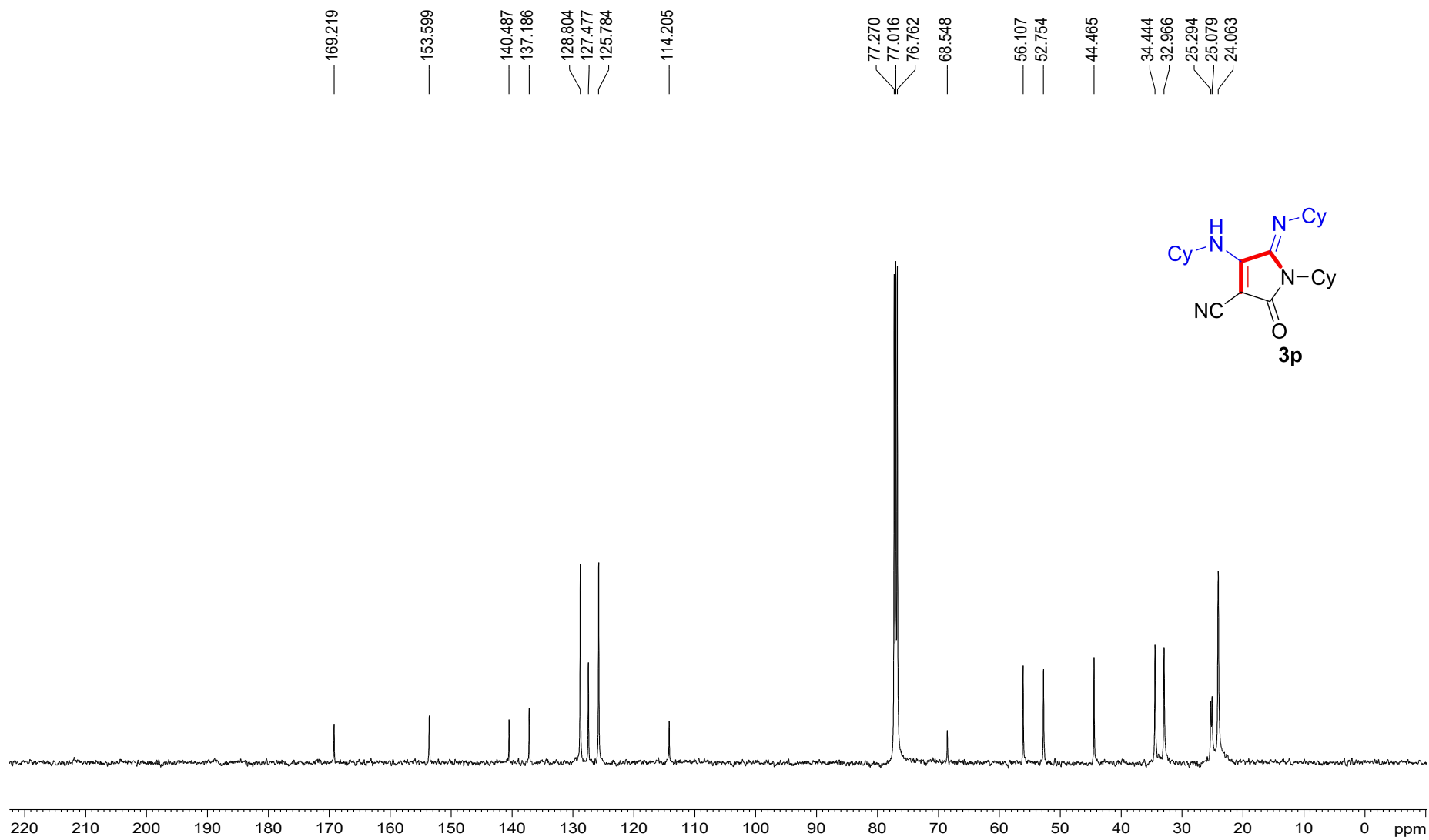


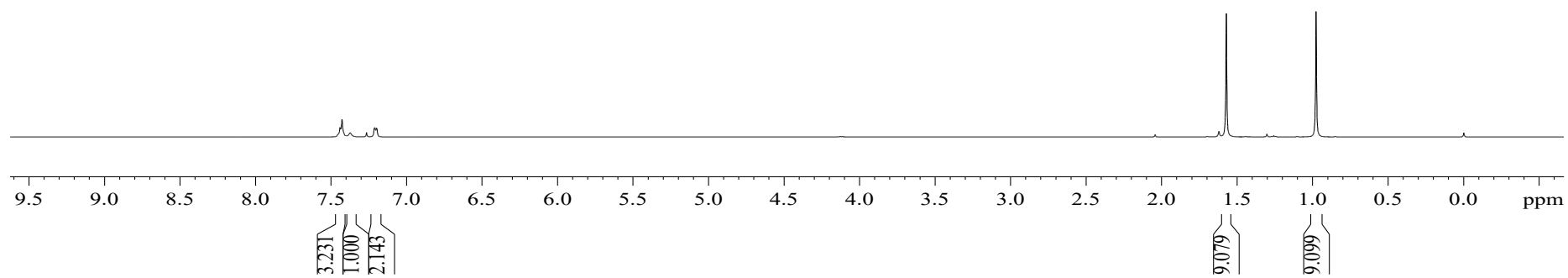
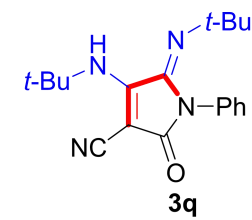
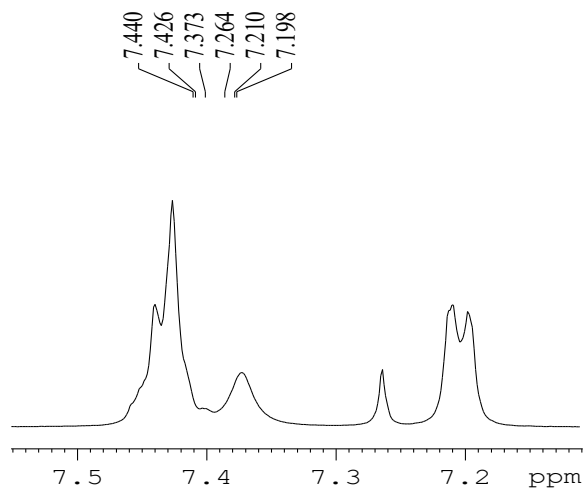


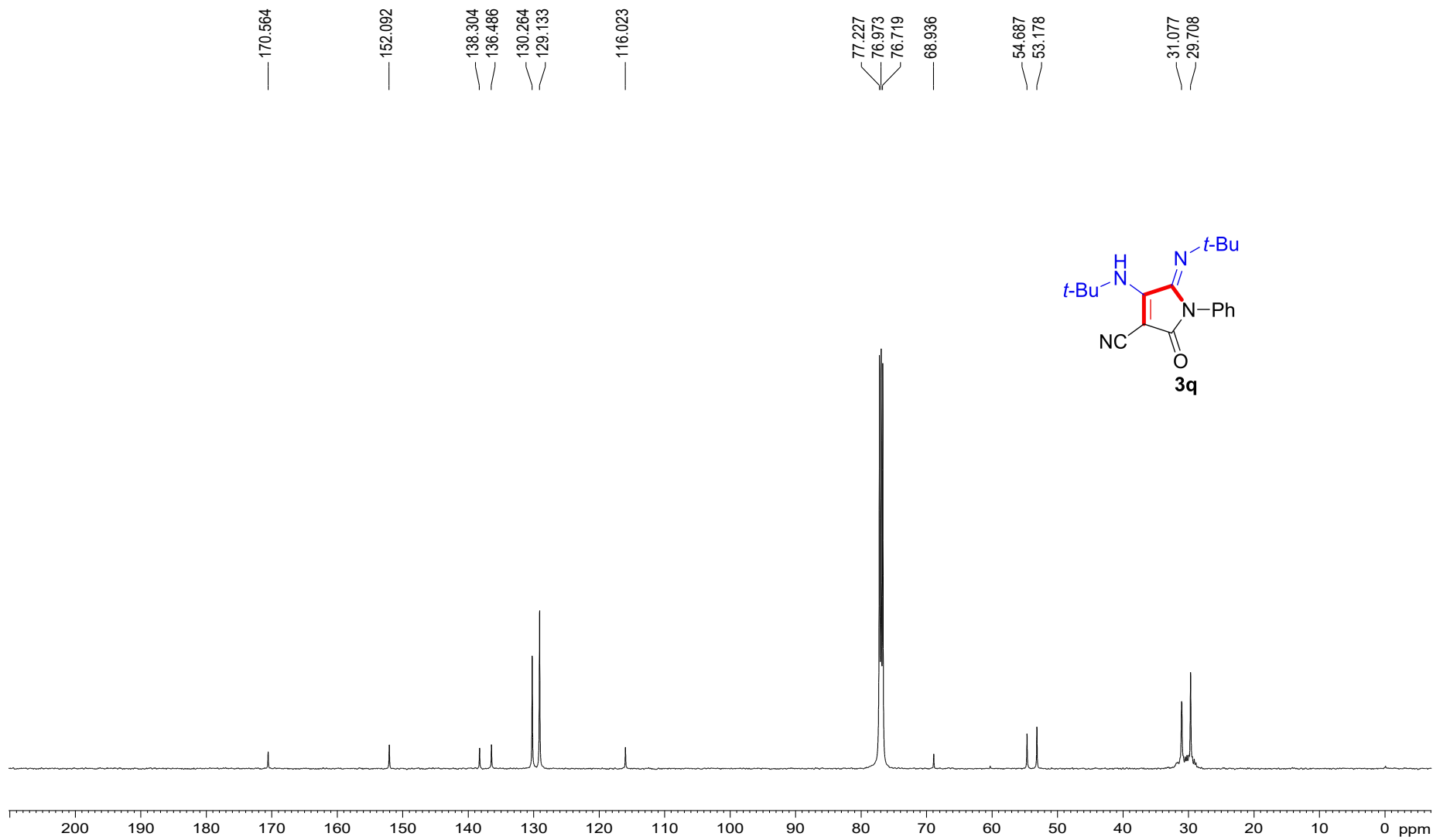


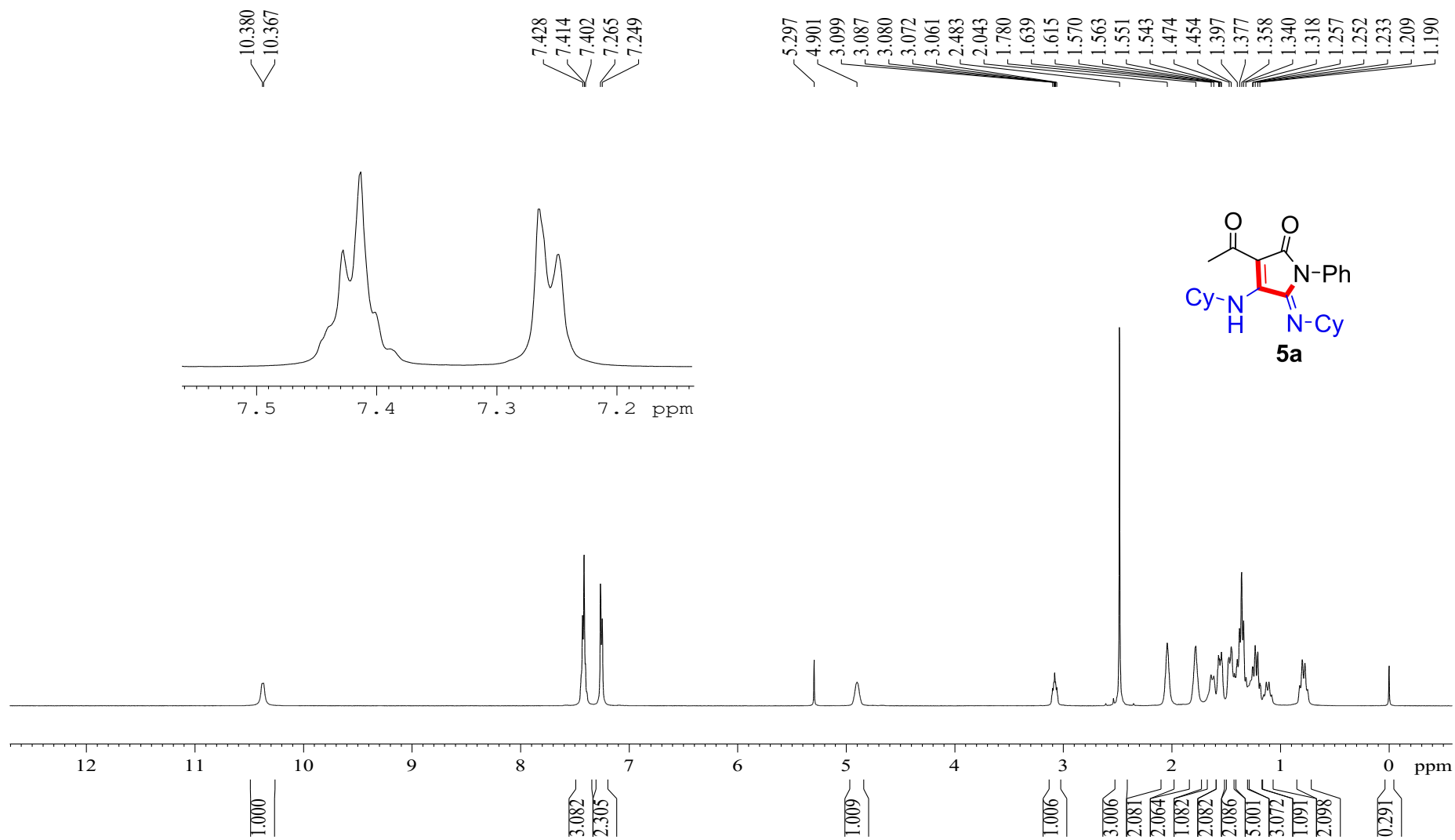


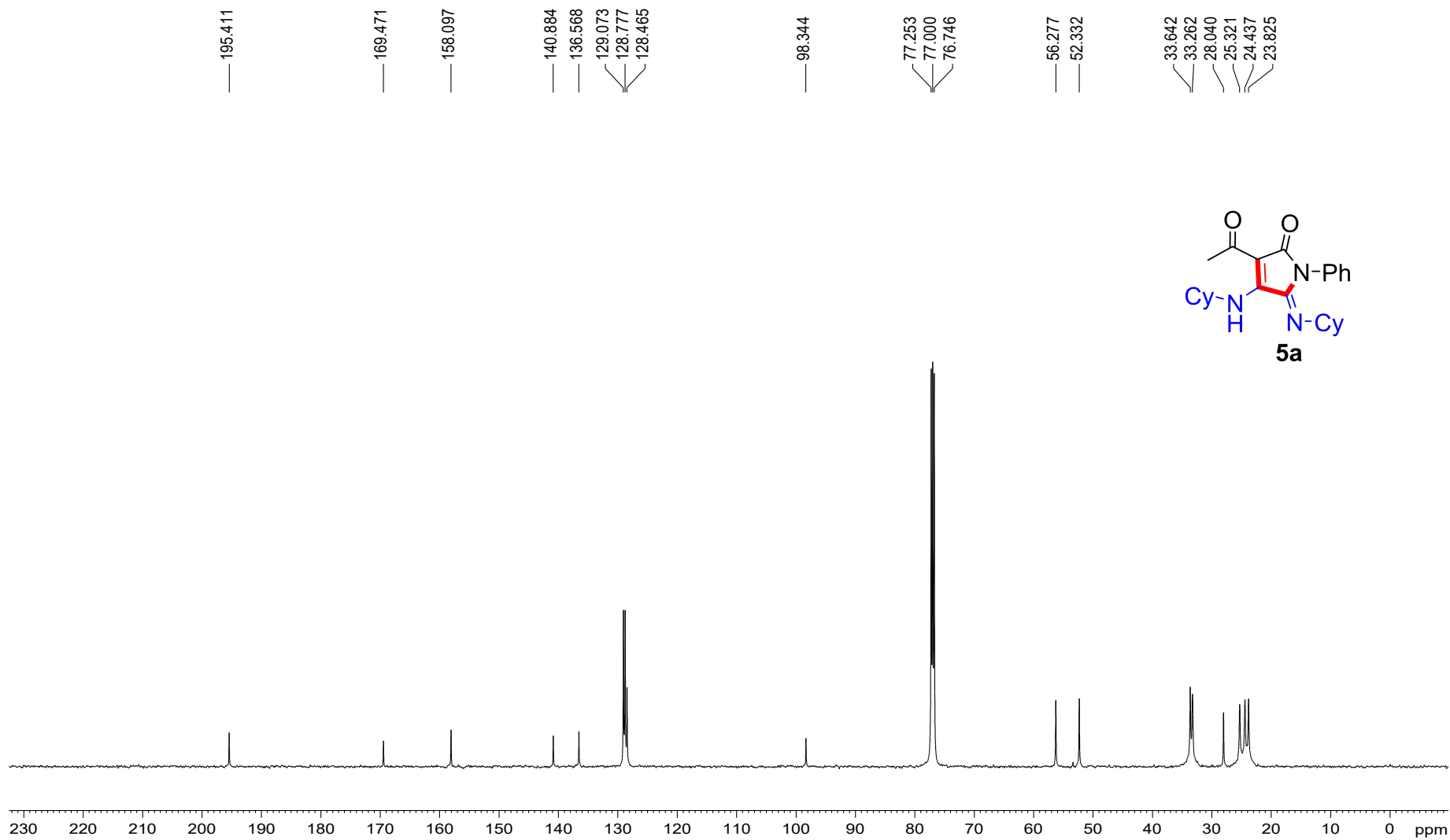


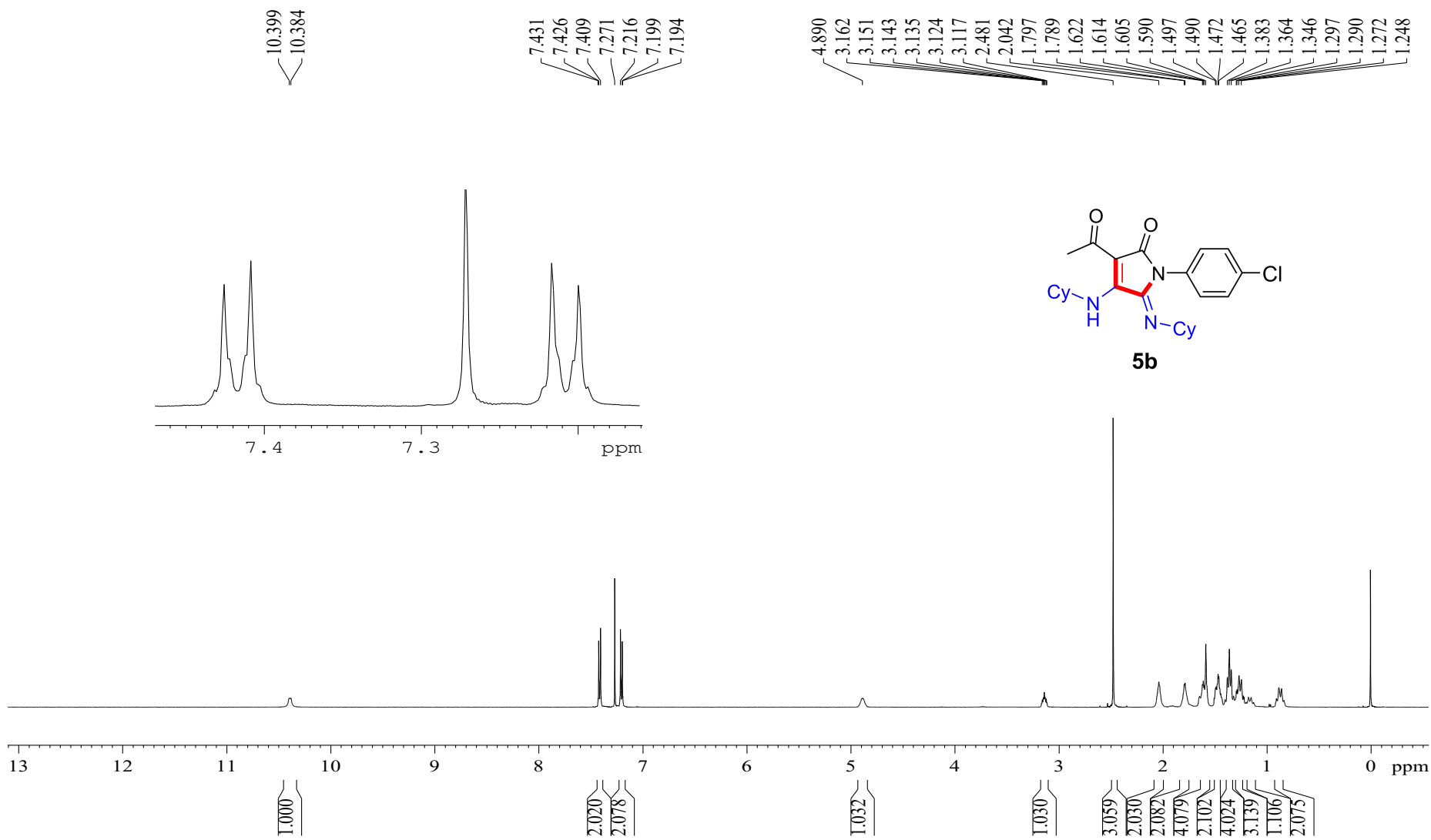


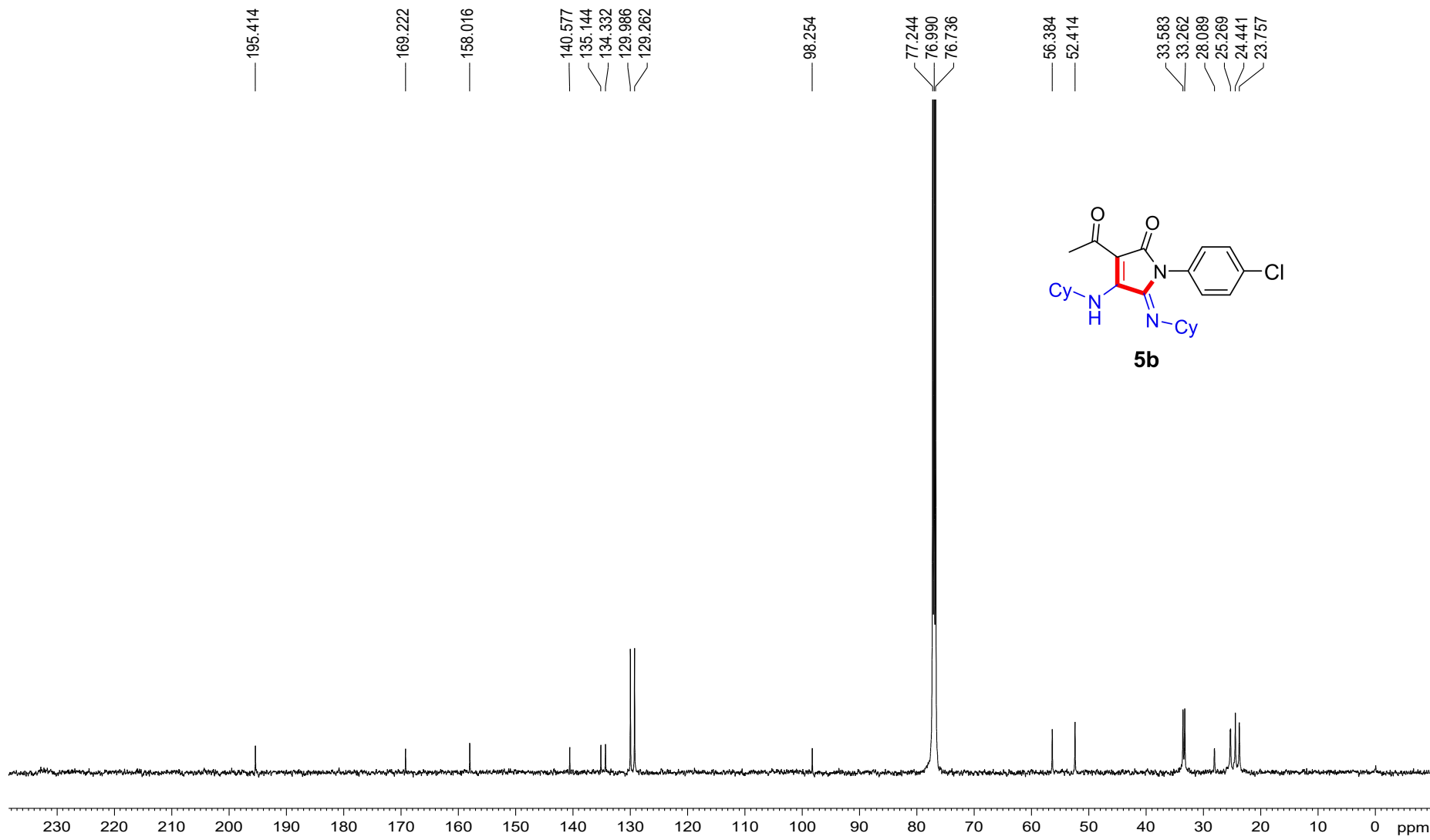


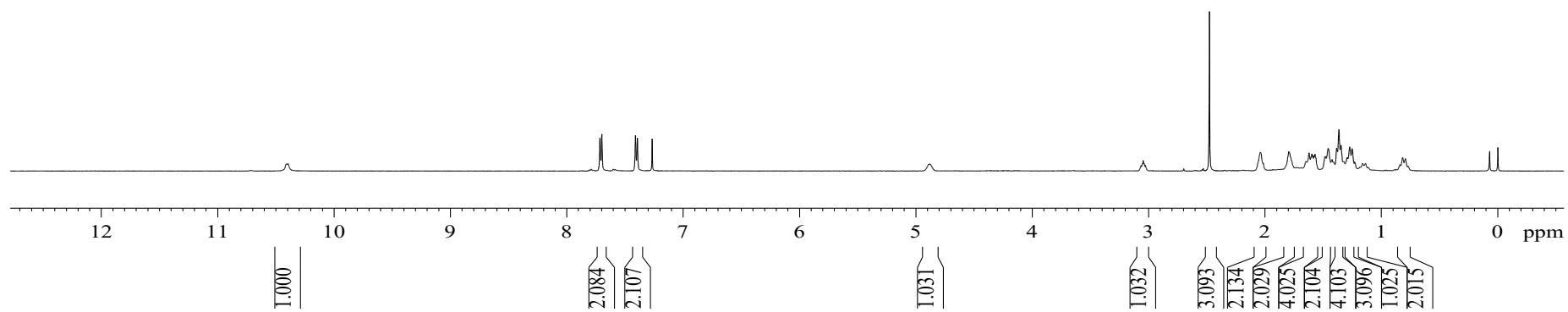
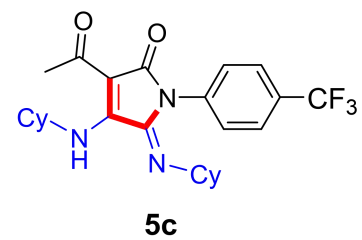
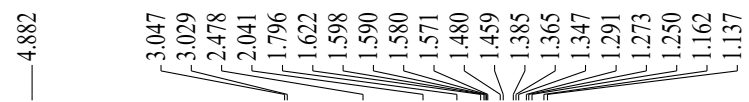
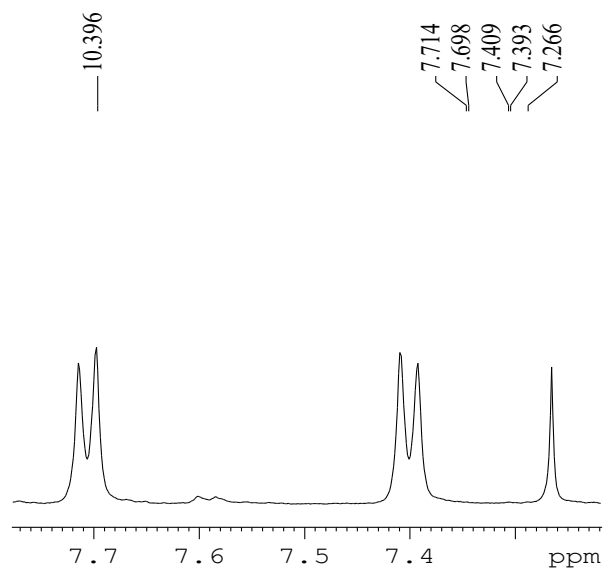


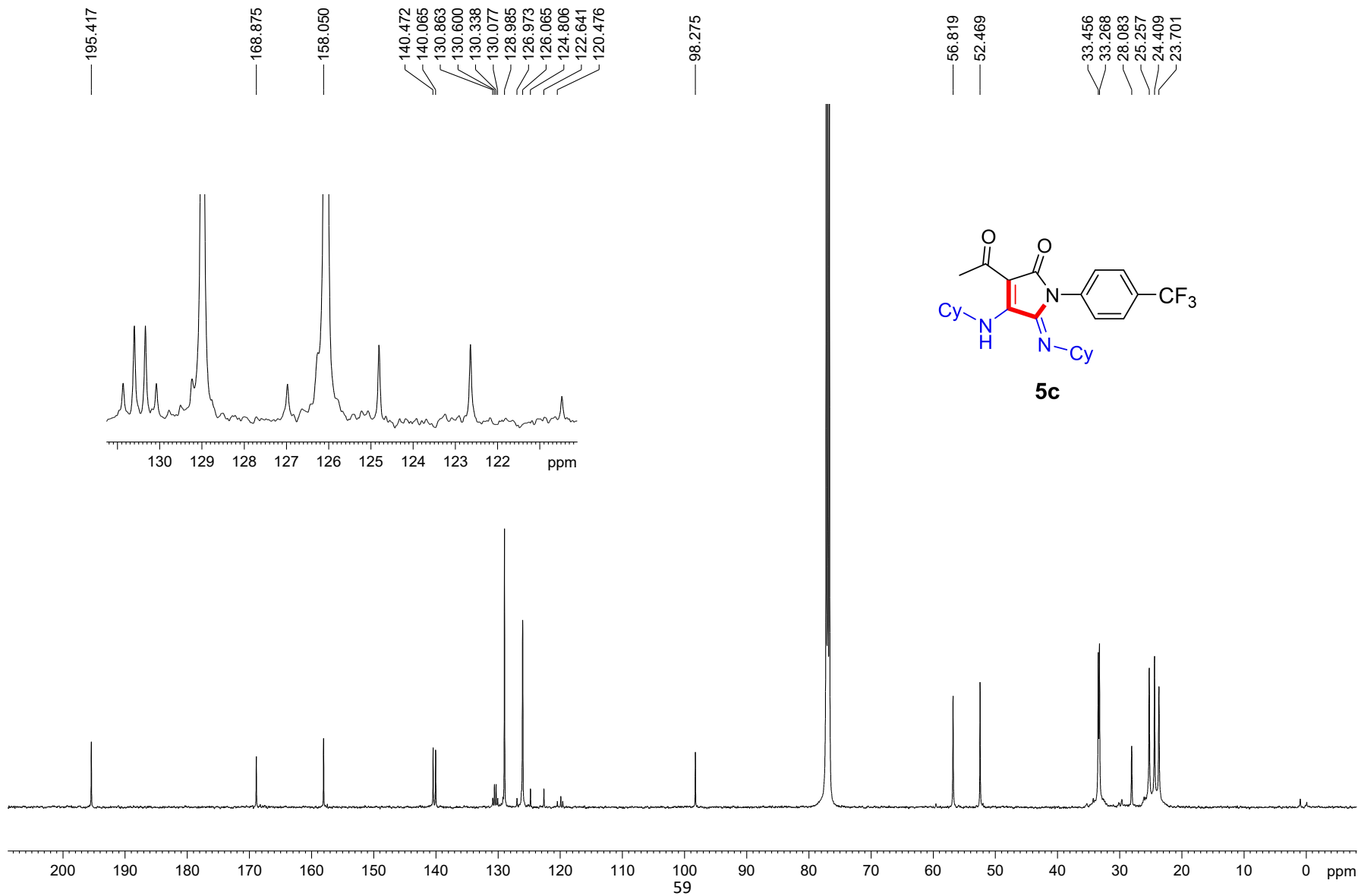


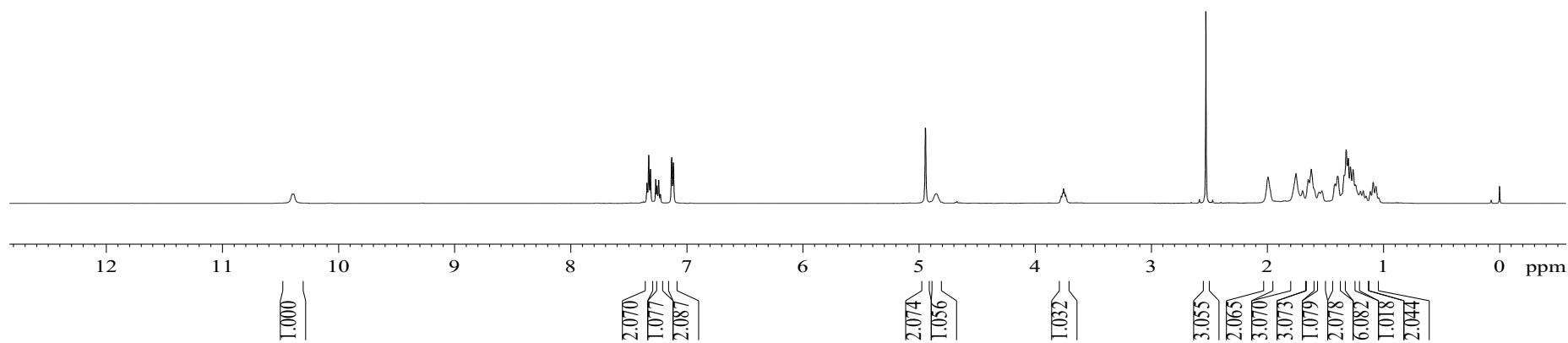
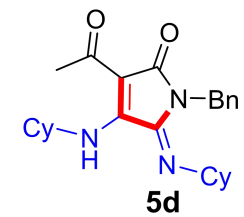
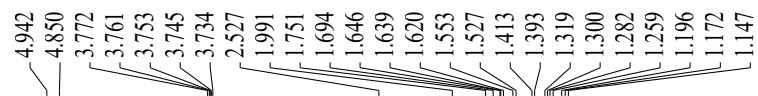
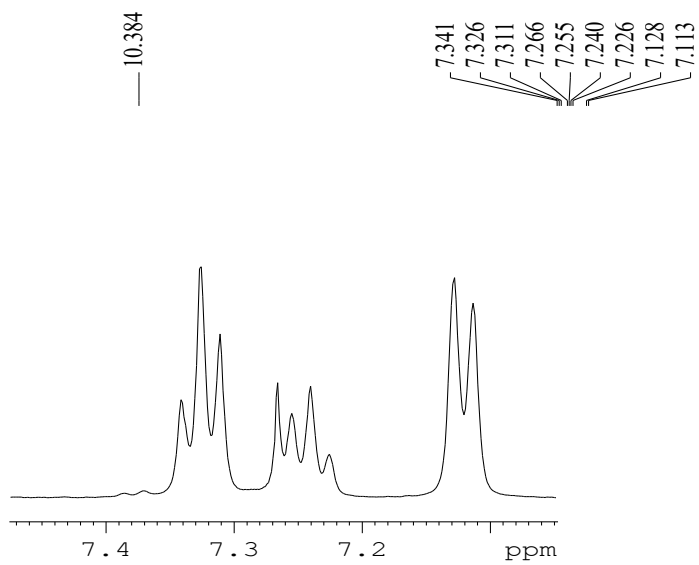


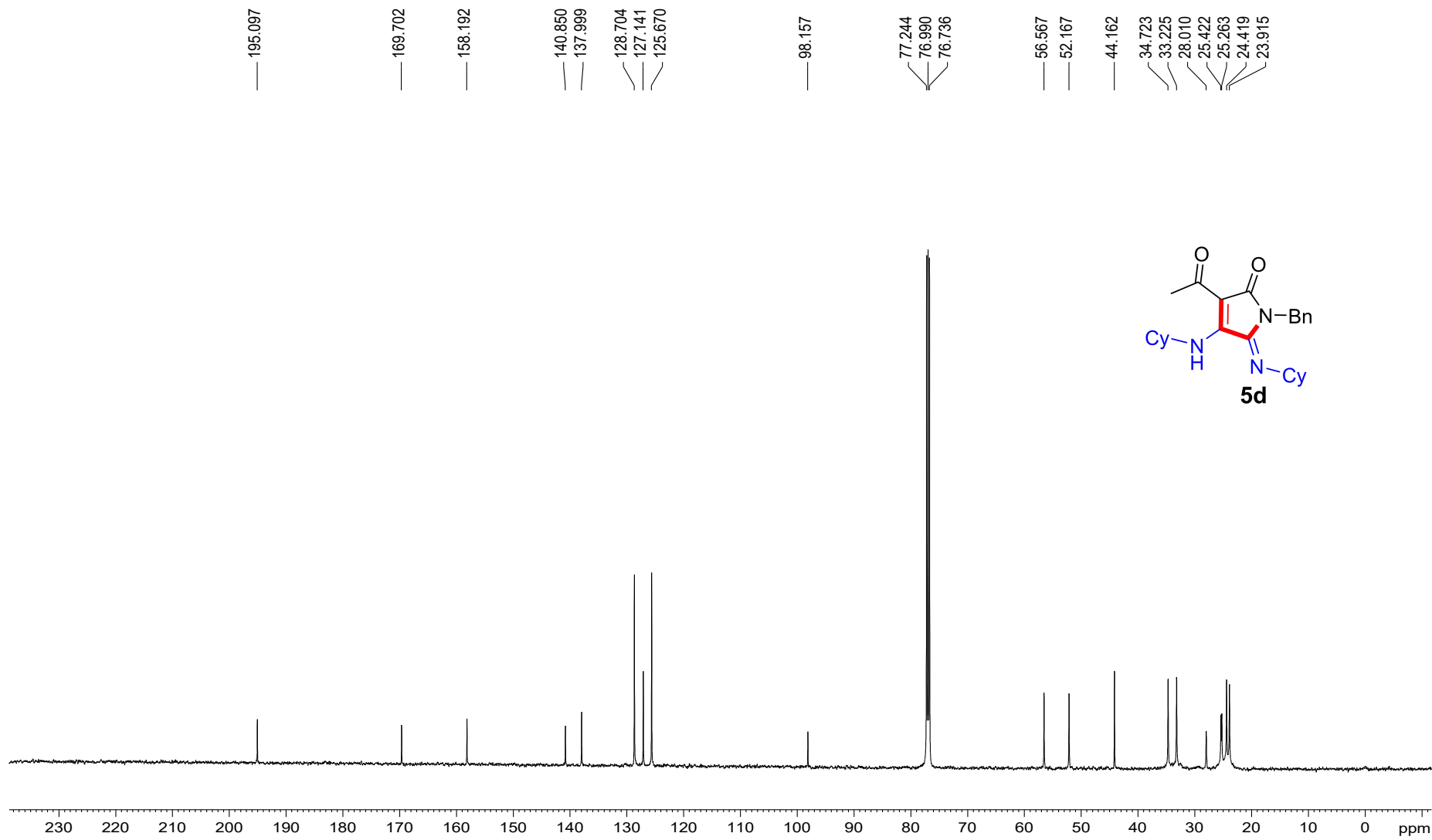


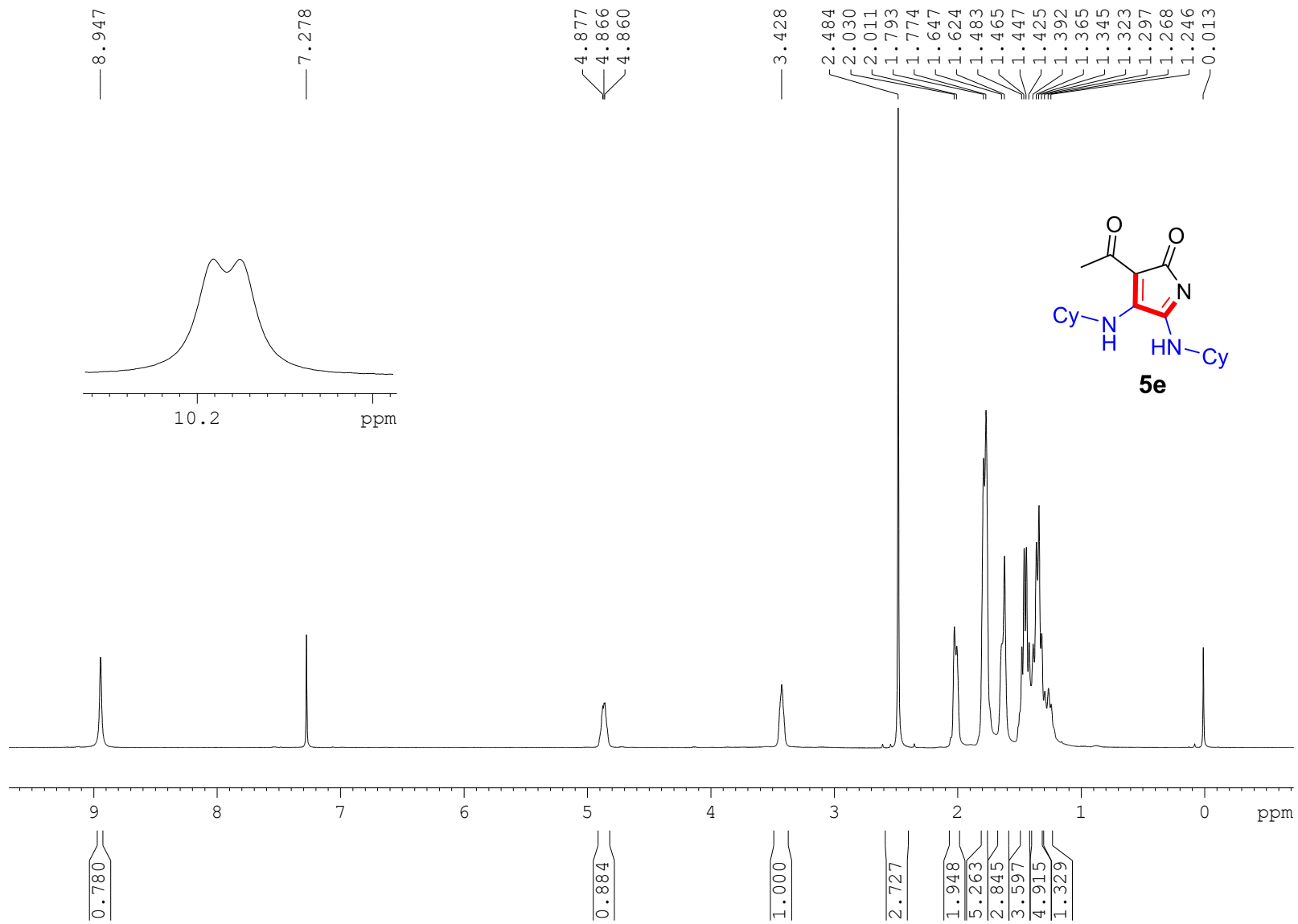


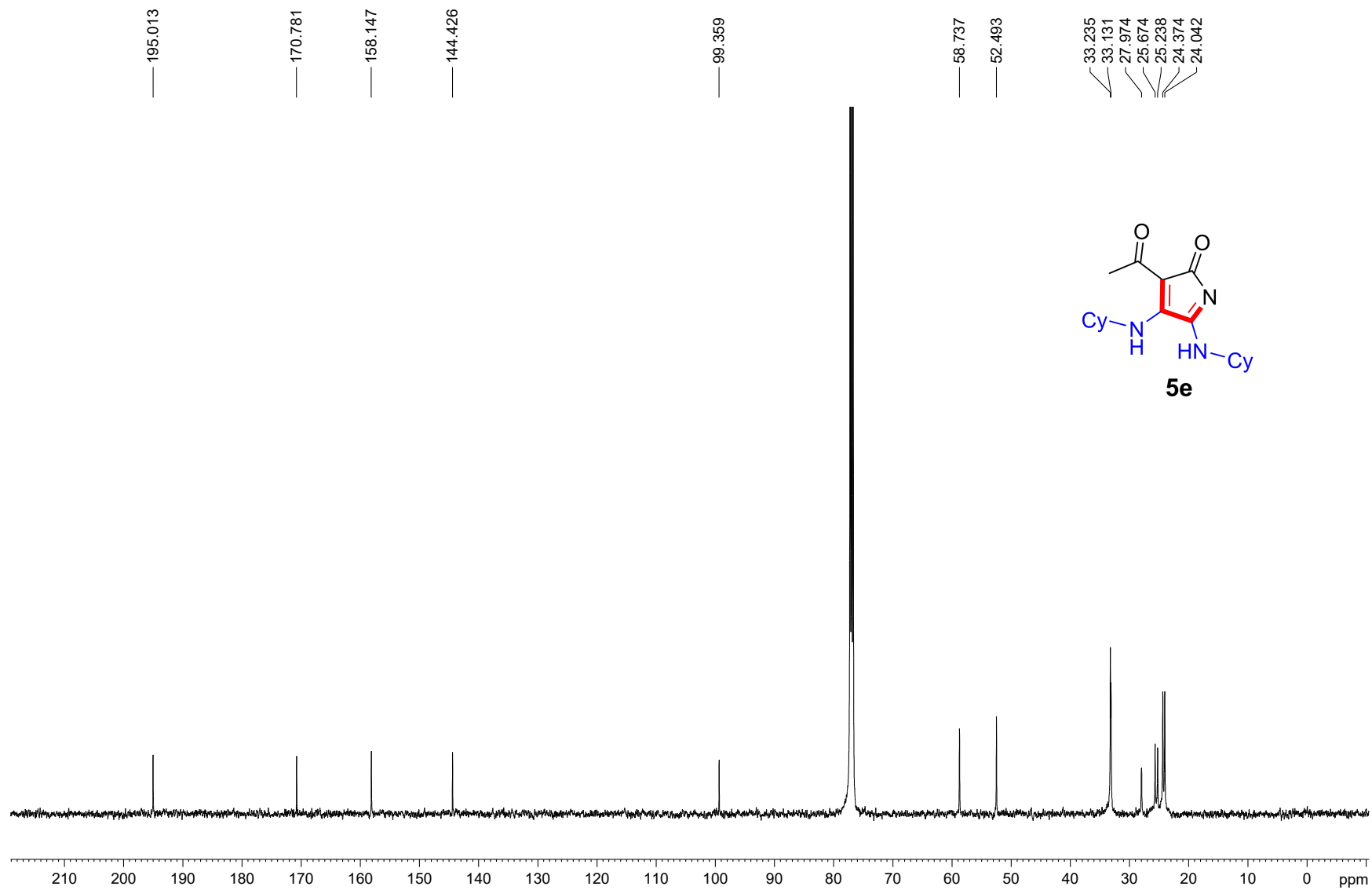


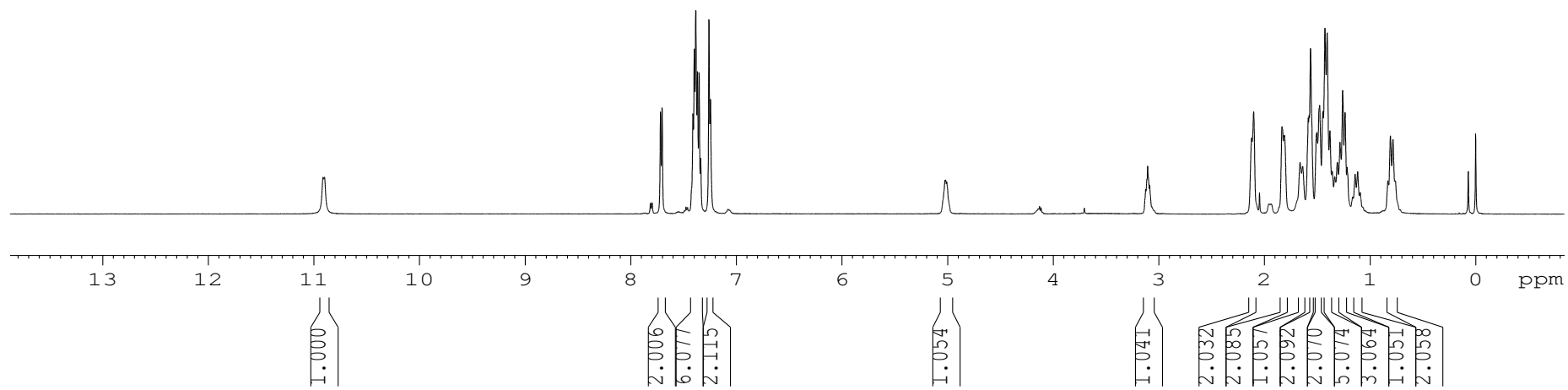
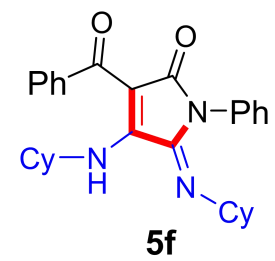
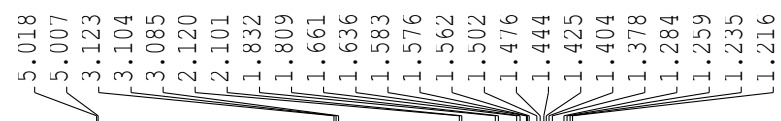
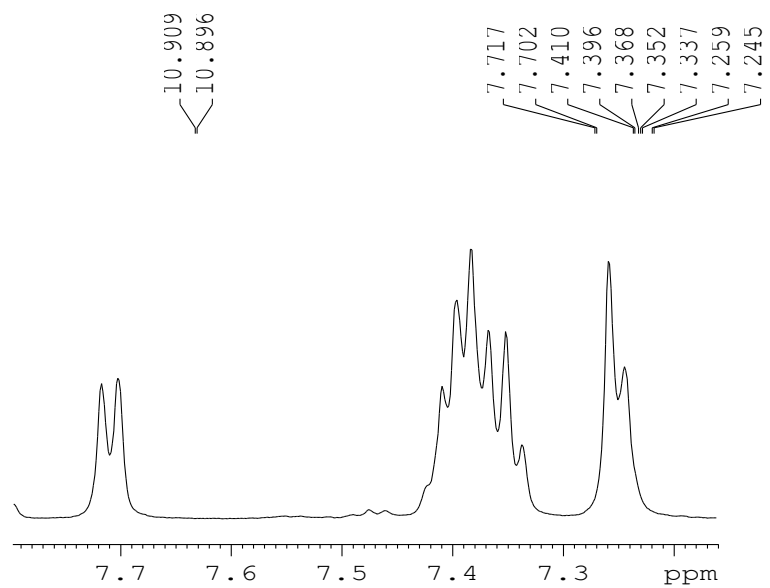


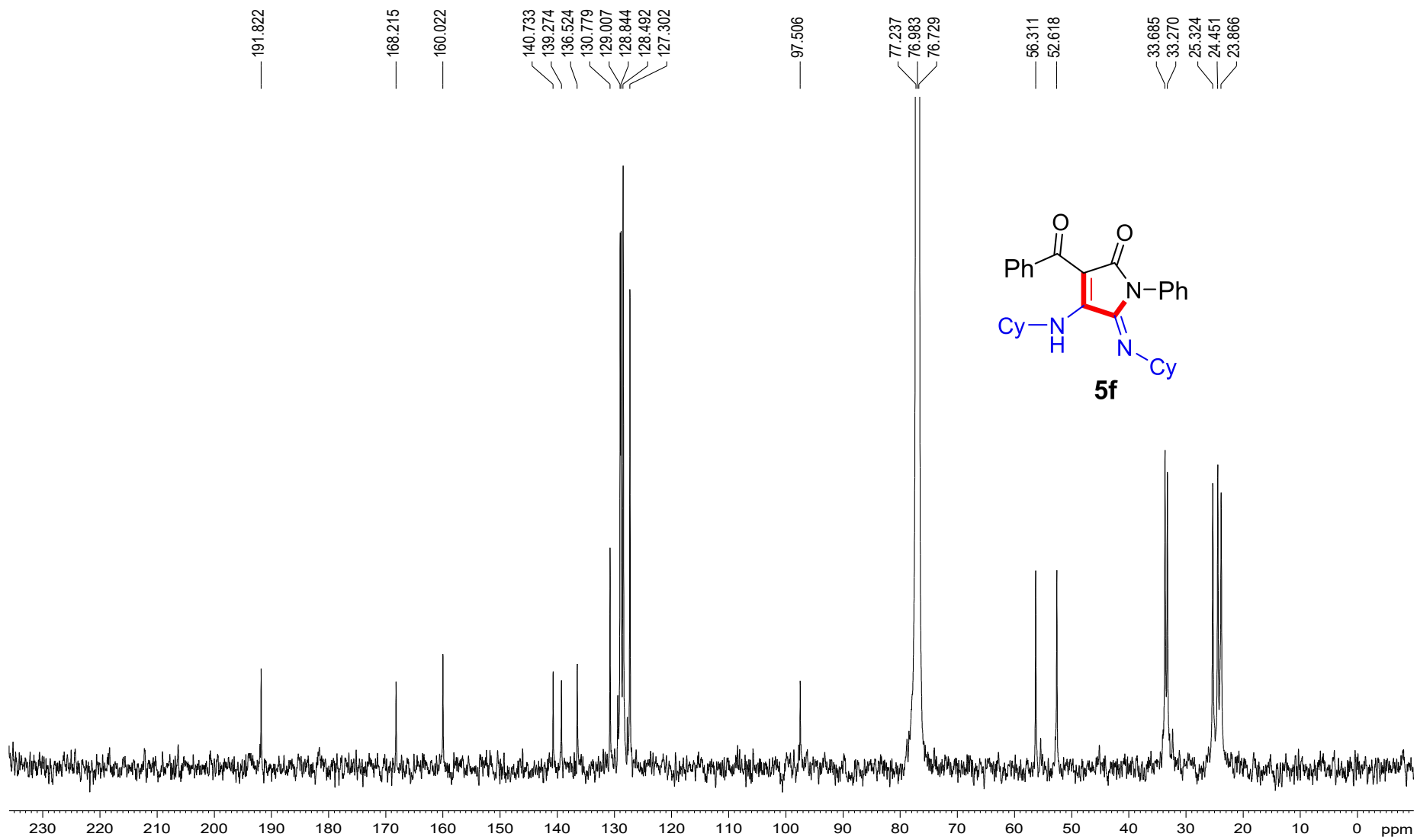


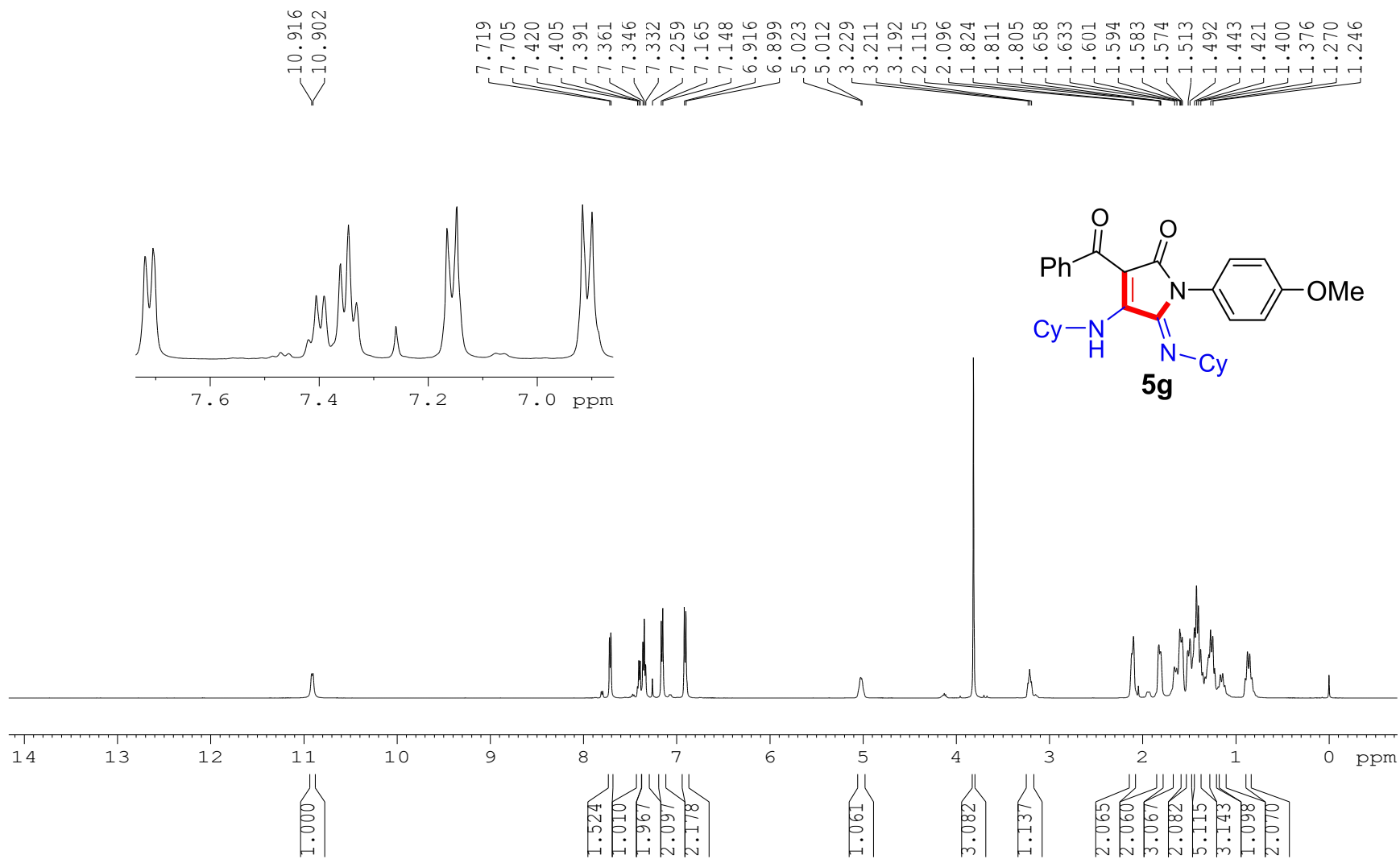


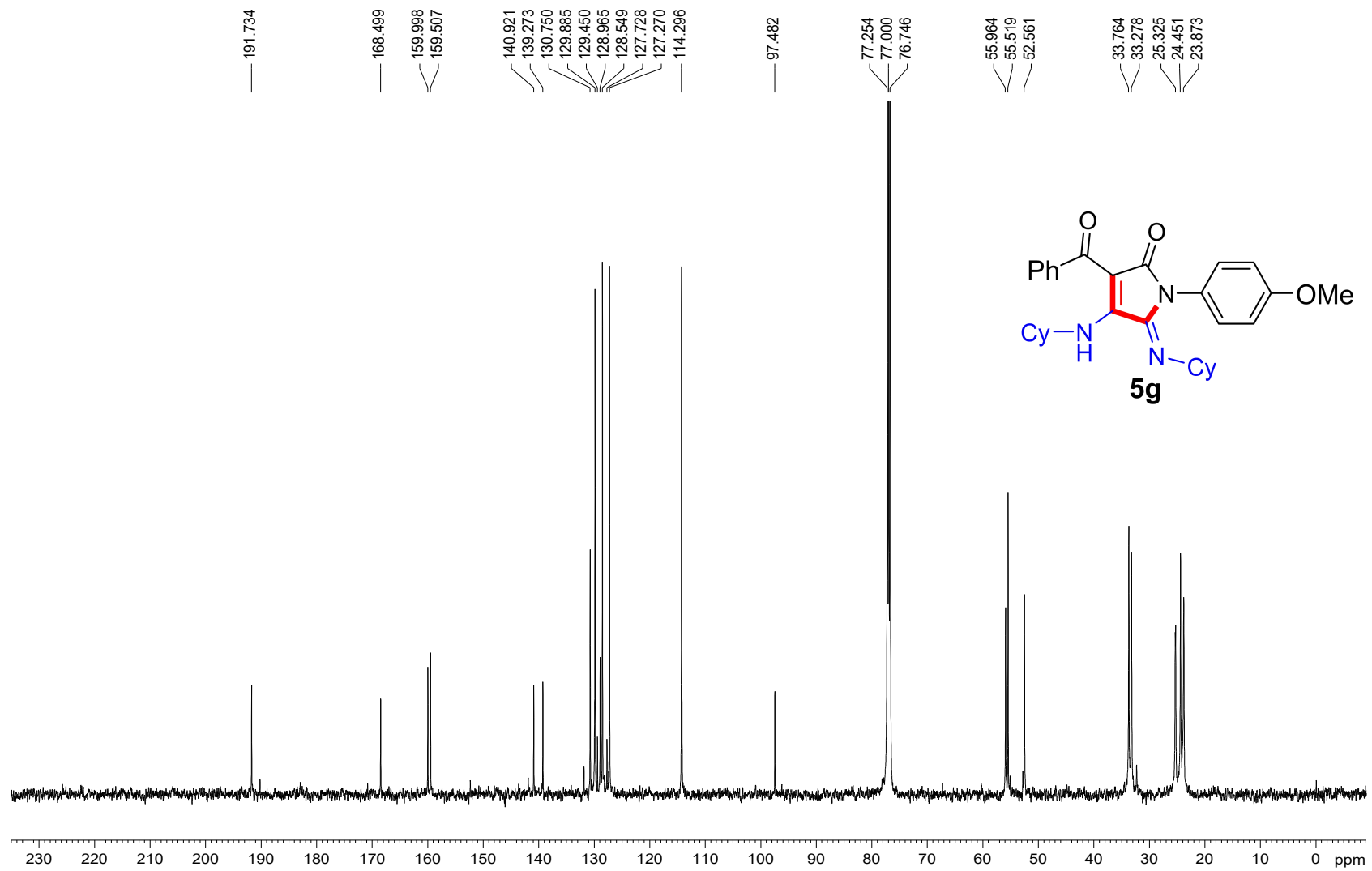












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