

## Supporting information

### **Molecular-iodine catalyzed selective construction of cyclopenta[*b*]indoles from indoles and acetone: a green gateway to indole-fused cycles**

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# 1. Copies of $^1\text{H}$ and $^{13}\text{C}$ NMR spectra

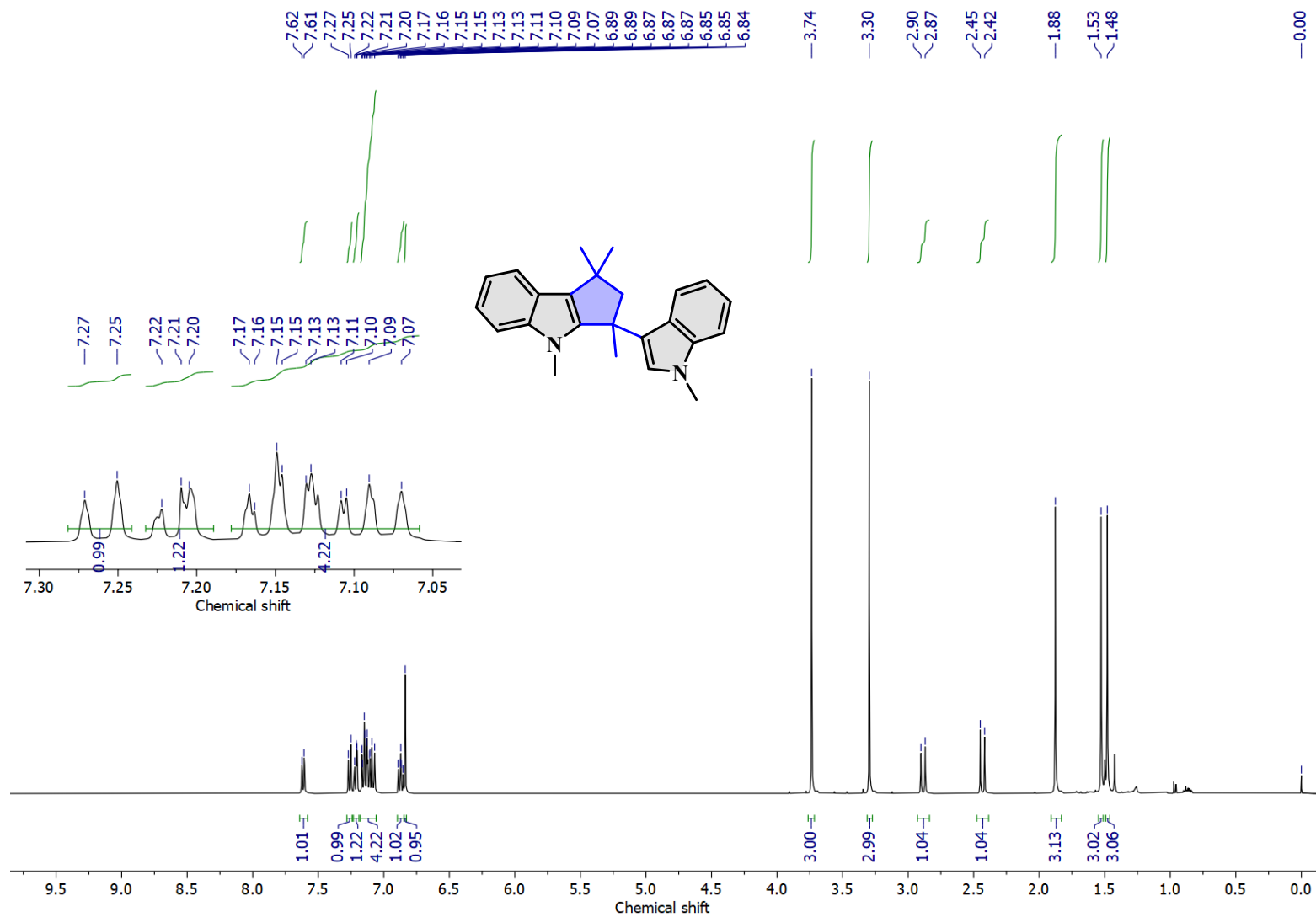


Fig. S1 400 MHz  $^1\text{H}$  NMR spectrum of (2a)<sup>1</sup> in  $\text{CDCl}_3$

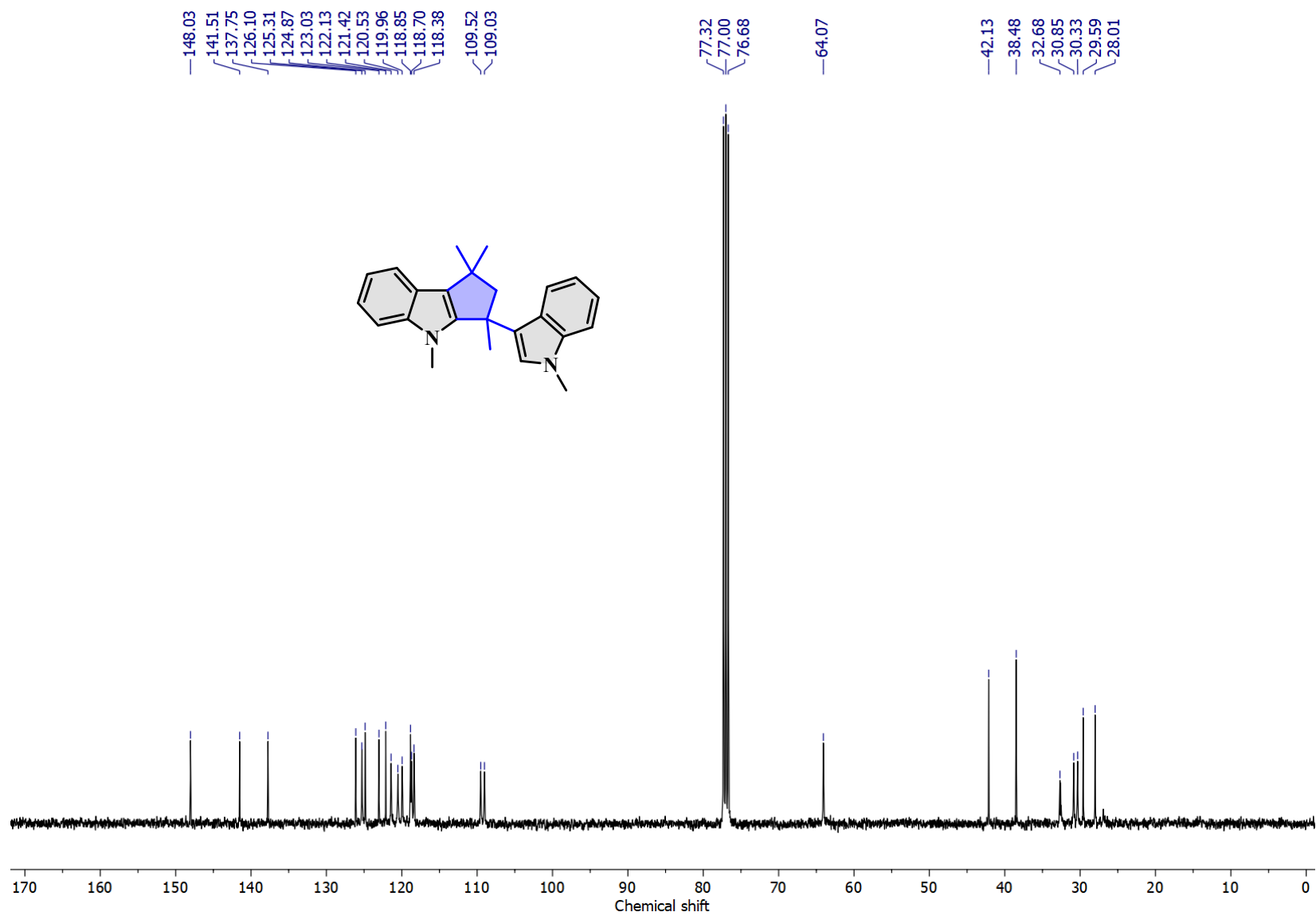


Fig. S2 100 MHz <sup>13</sup>C NMR spectrum of (2a)<sup>1</sup> in CDCl<sub>3</sub>

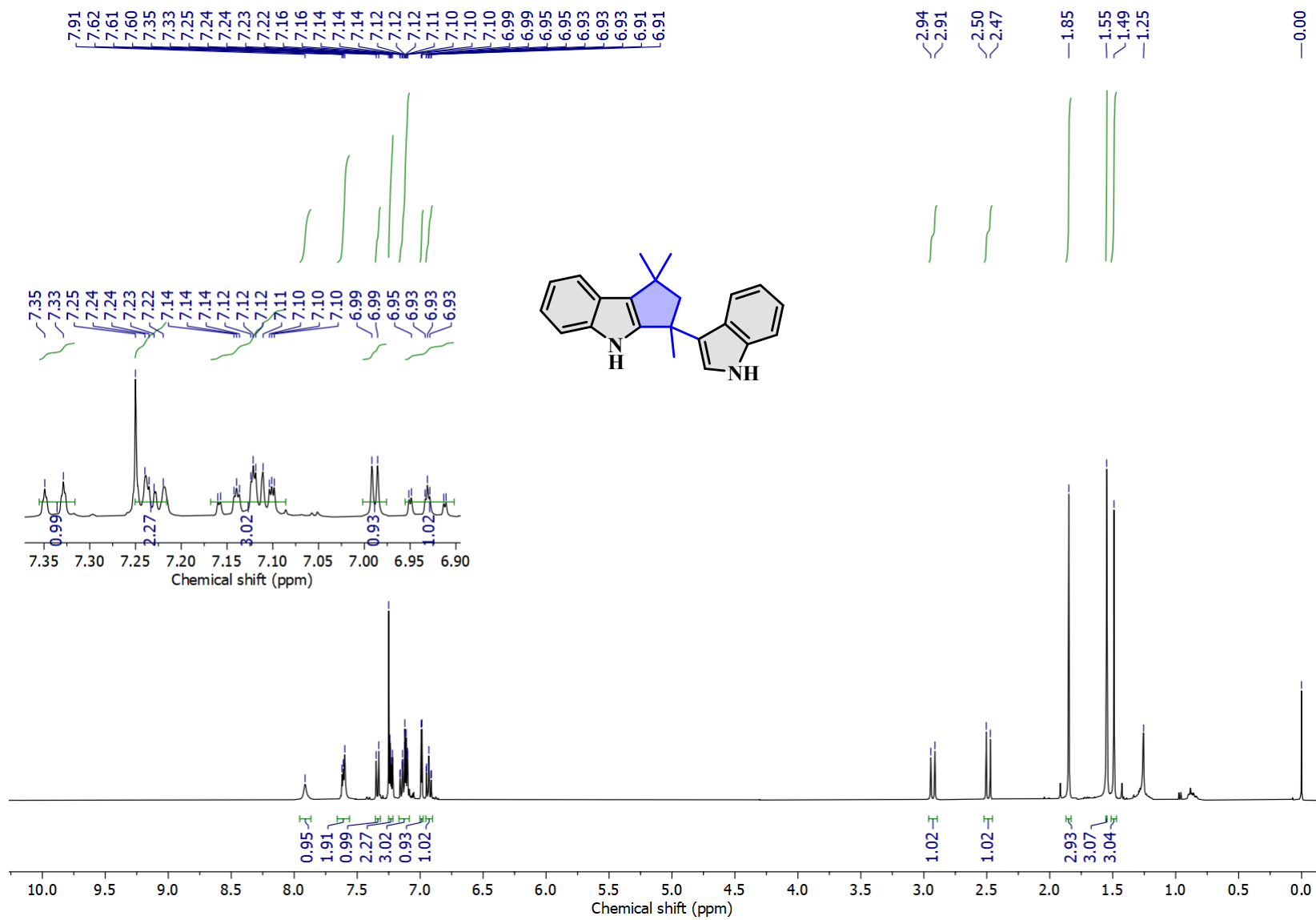


Fig. S3 400 MHz <sup>1</sup>H NMR spectrum of (2b)<sup>1</sup> in CDCl<sub>3</sub>

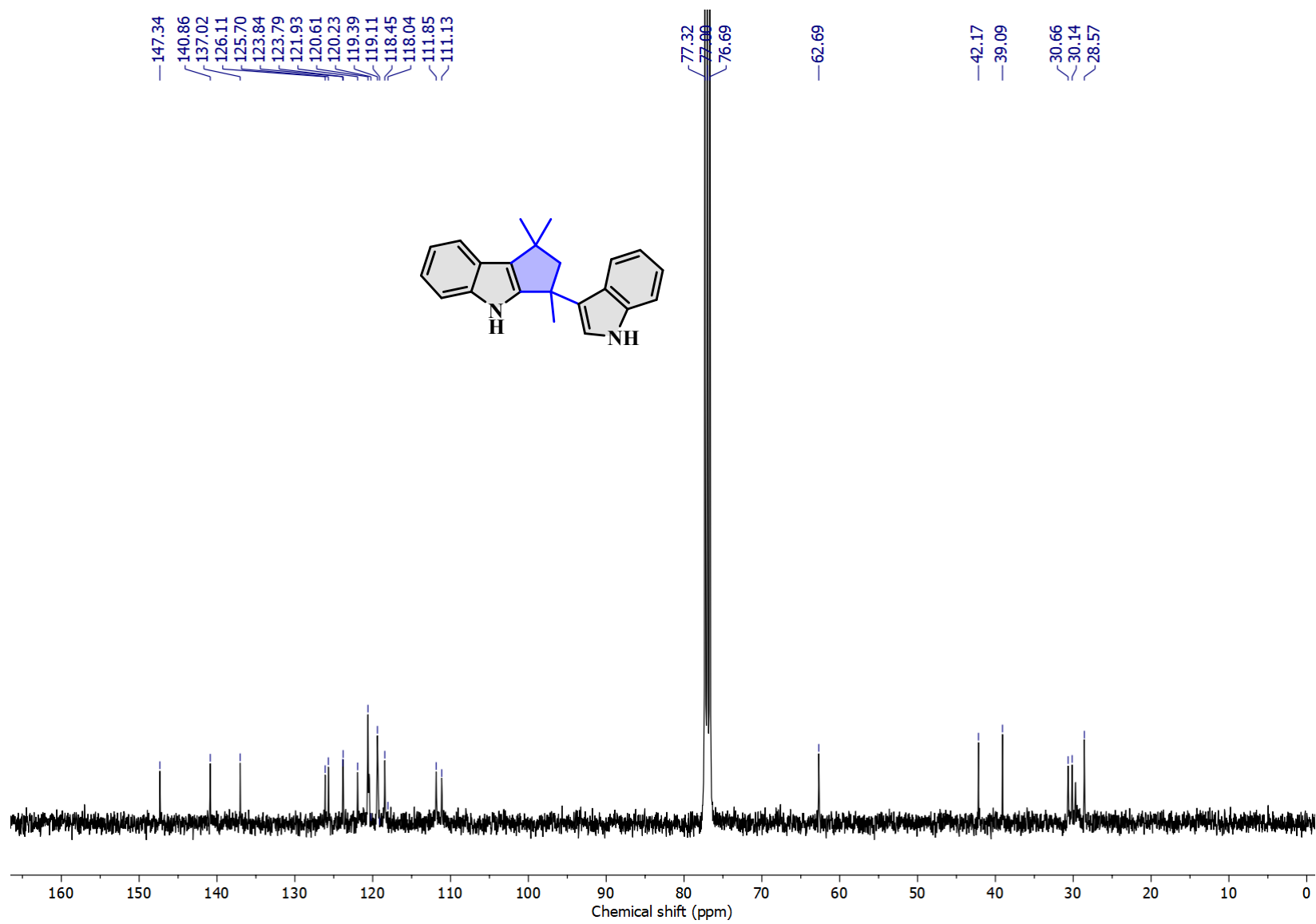


Fig. S4 100 MHz  $^{13}\text{C}$  NMR spectrum of **(2b)**<sup>1</sup> in  $\text{CDCl}_3$

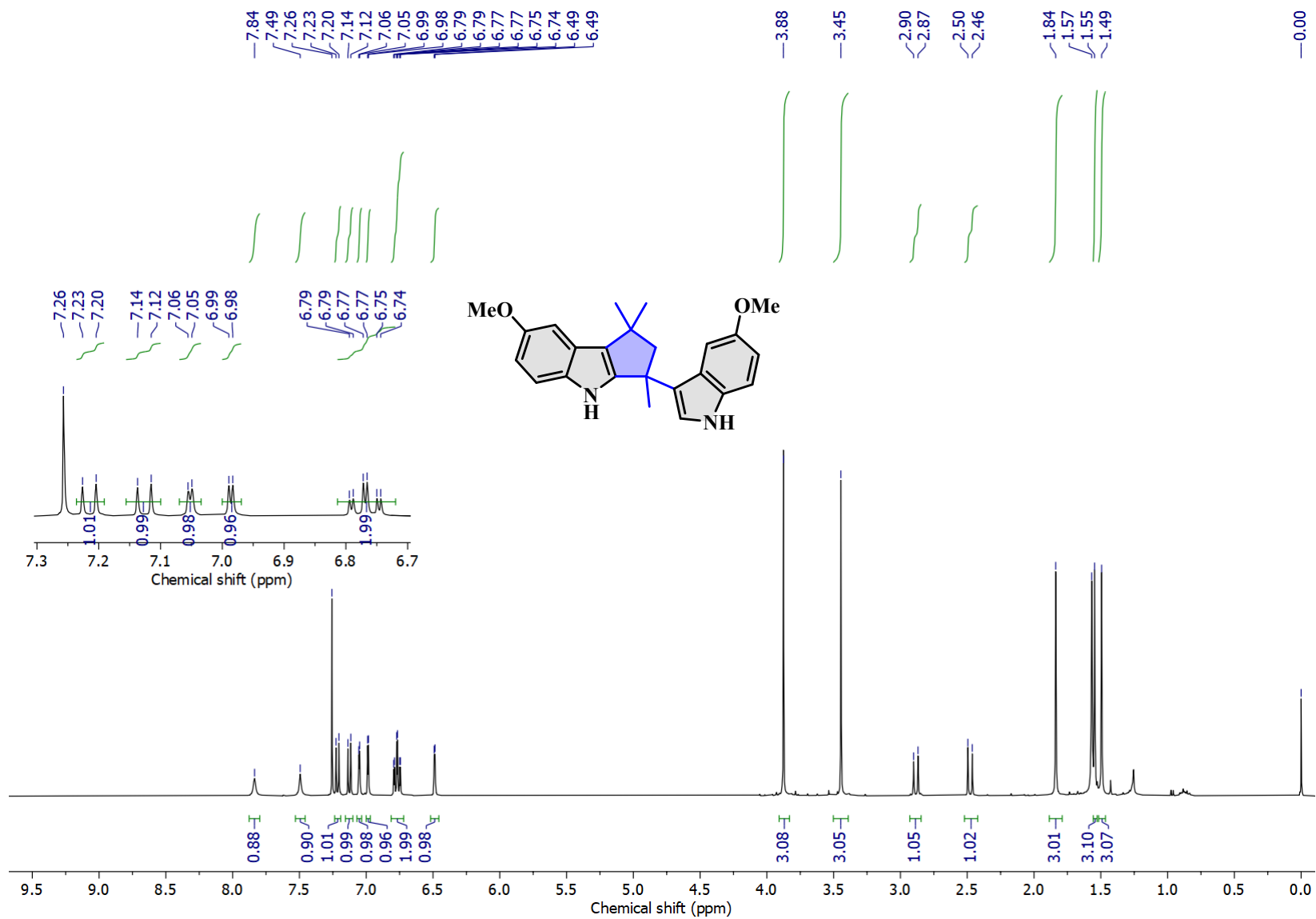


Fig. S5 400 MHz <sup>1</sup>H NMR spectrum of **(2c)<sup>2</sup>** in CDCl<sub>3</sub>

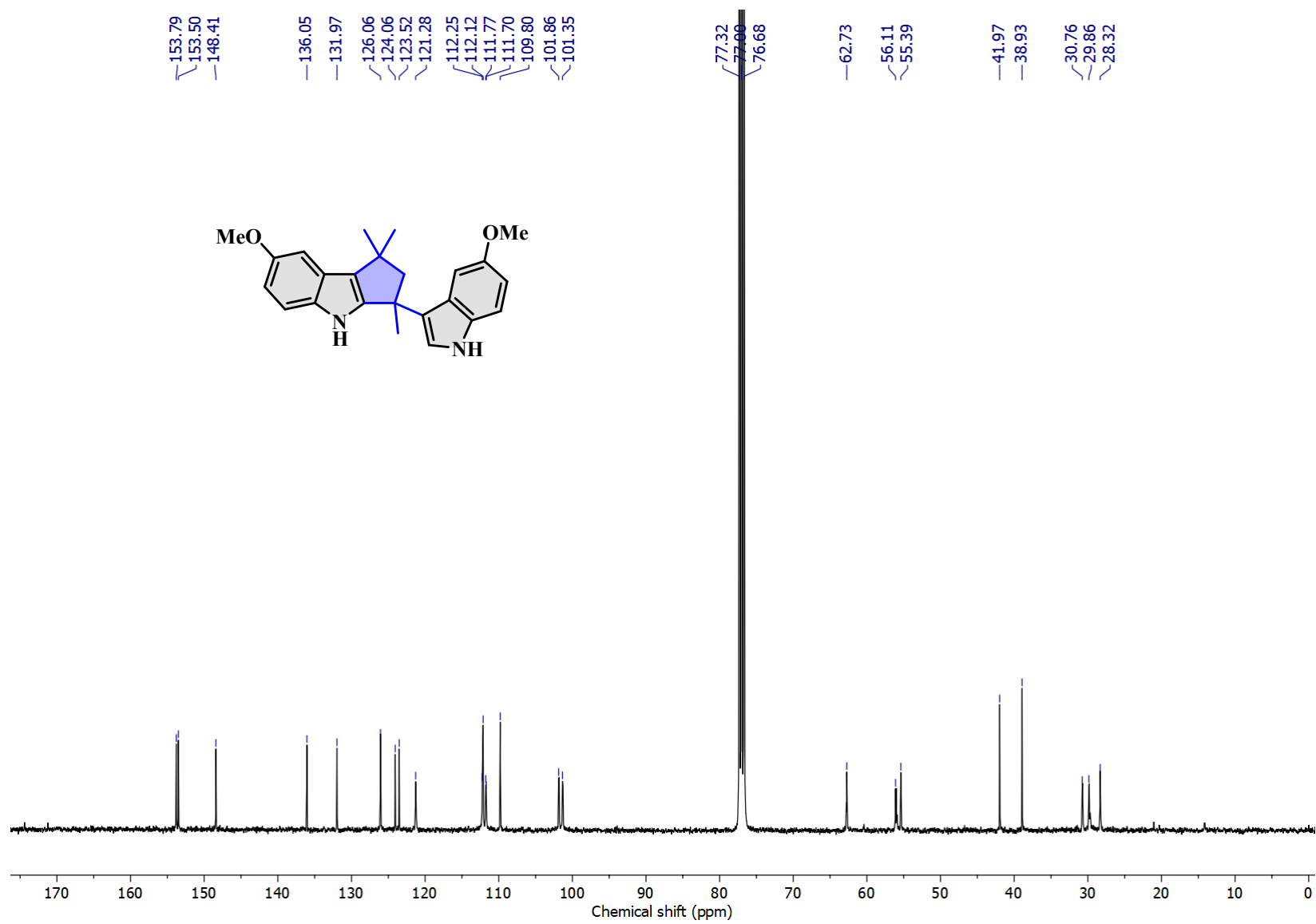


Fig. S6 100 MHz <sup>13</sup>C NMR spectrum of **(2c)<sup>2</sup>** in CDCl<sub>3</sub>





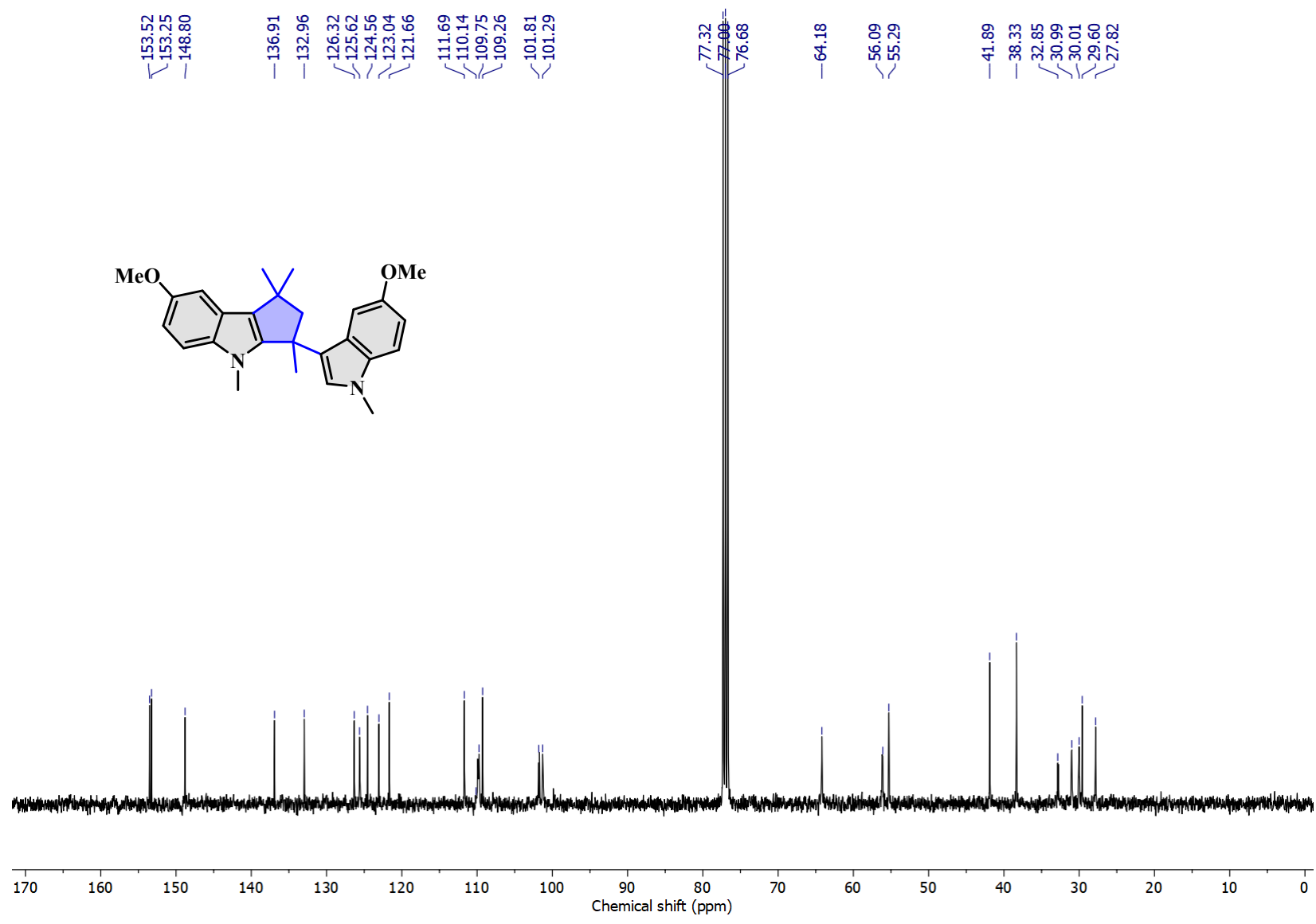
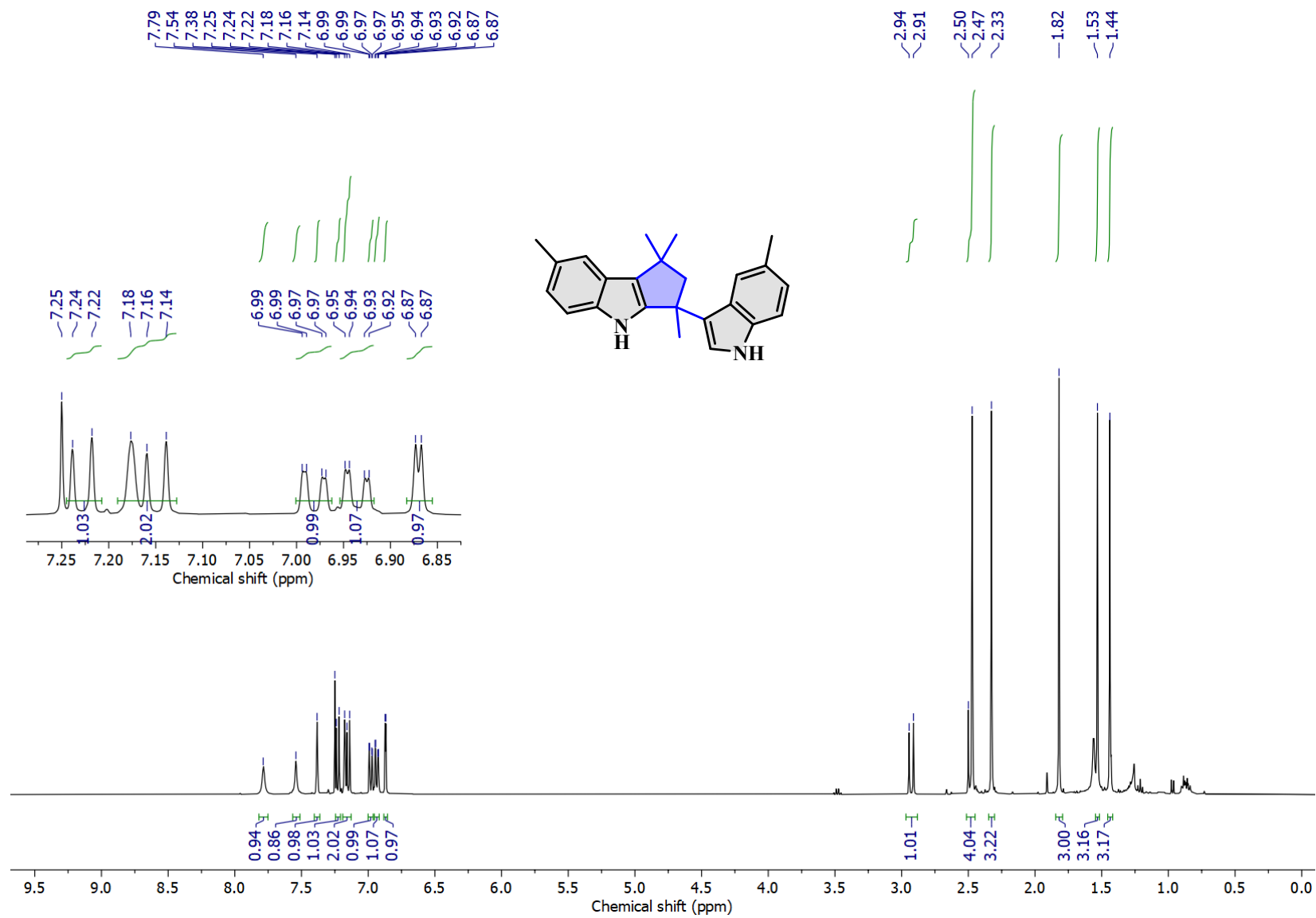


Fig. S8 100 MHz <sup>13</sup>C NMR spectrum of (2d)<sup>2</sup> in CDCl<sub>3</sub>



**Fig. S9** 400 MHz <sup>1</sup>H NMR spectrum of **2e** in CDCl<sub>3</sub>

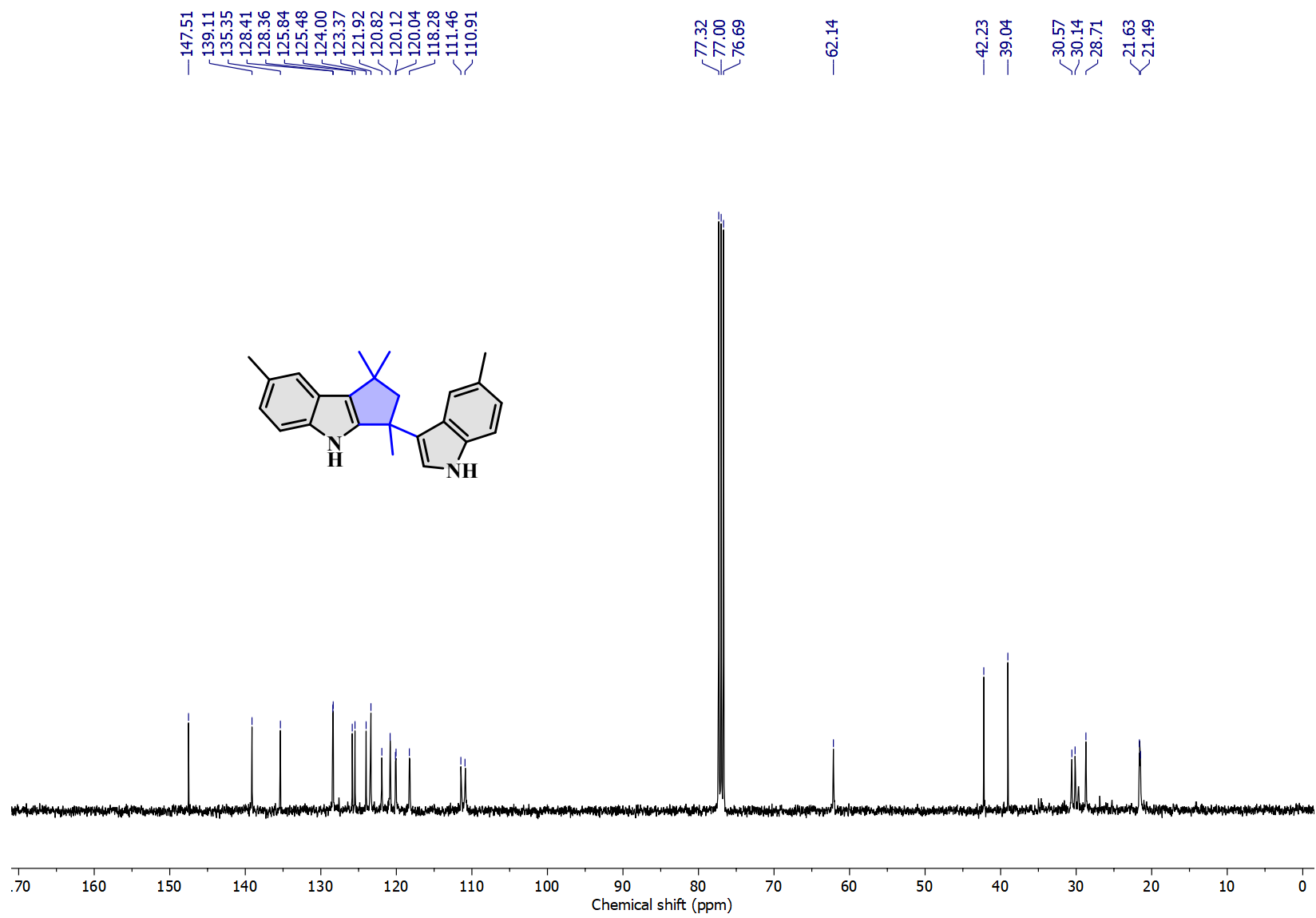


Fig. S10 100 MHz  $^{13}\text{C}$  NMR spectrum of **2e** in  $\text{CDCl}_3$

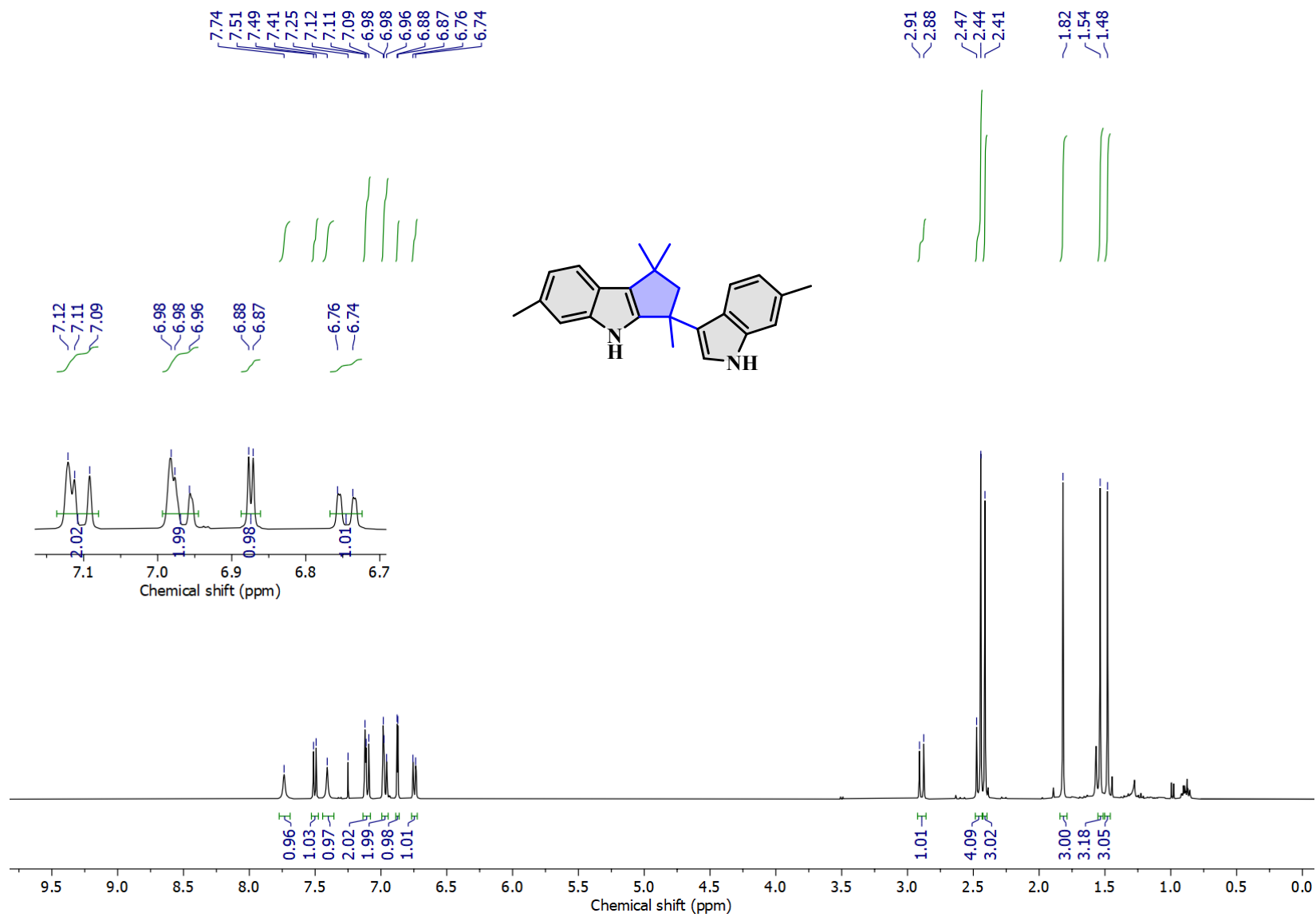
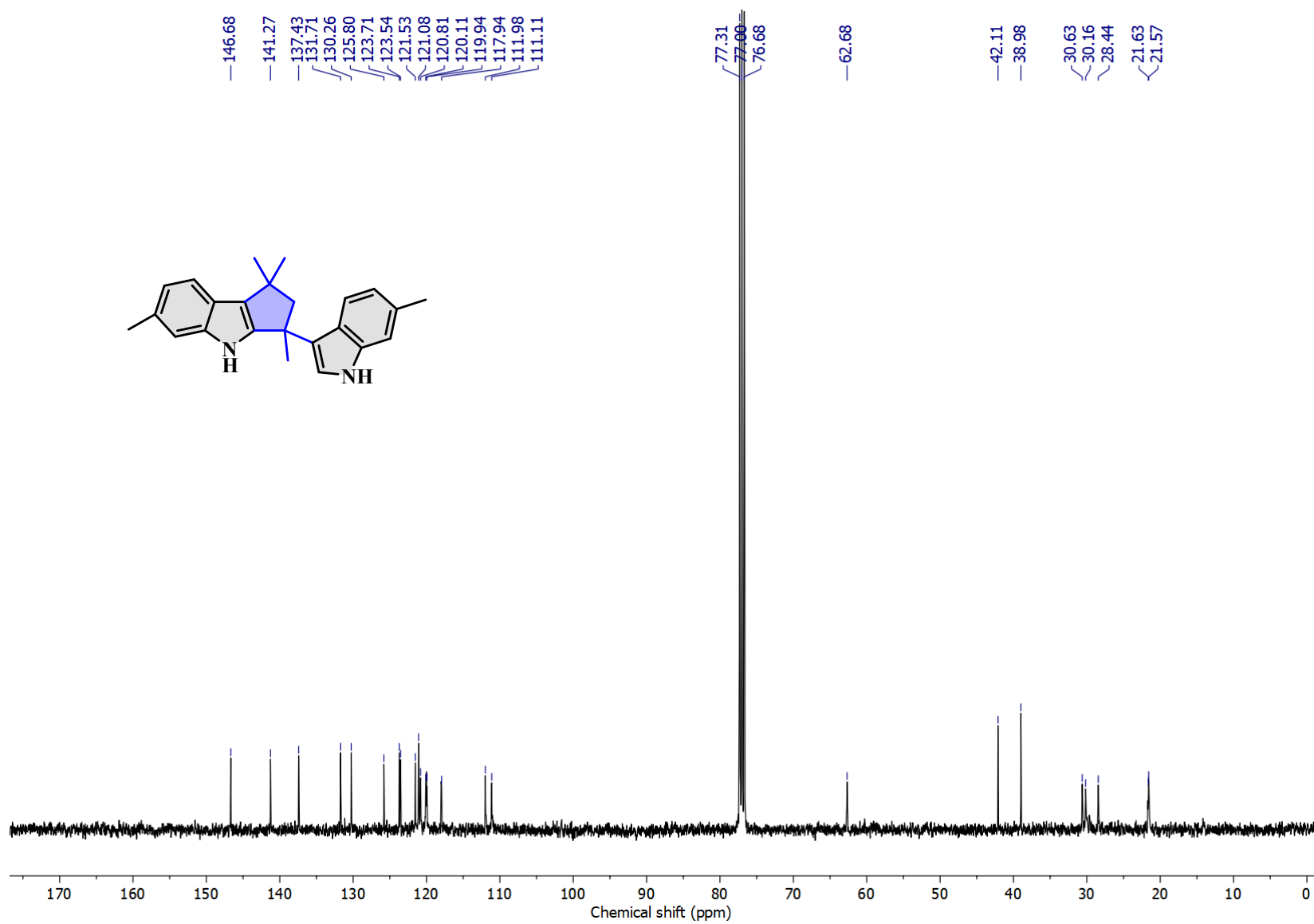


Fig. S11 400 MHz <sup>1</sup>H NMR spectrum of **2f** in CDCl<sub>3</sub>



**Fig. S12** 100 MHz  $^{13}\text{C}$  NMR spectrum of **2f** in  $\text{CDCl}_3$

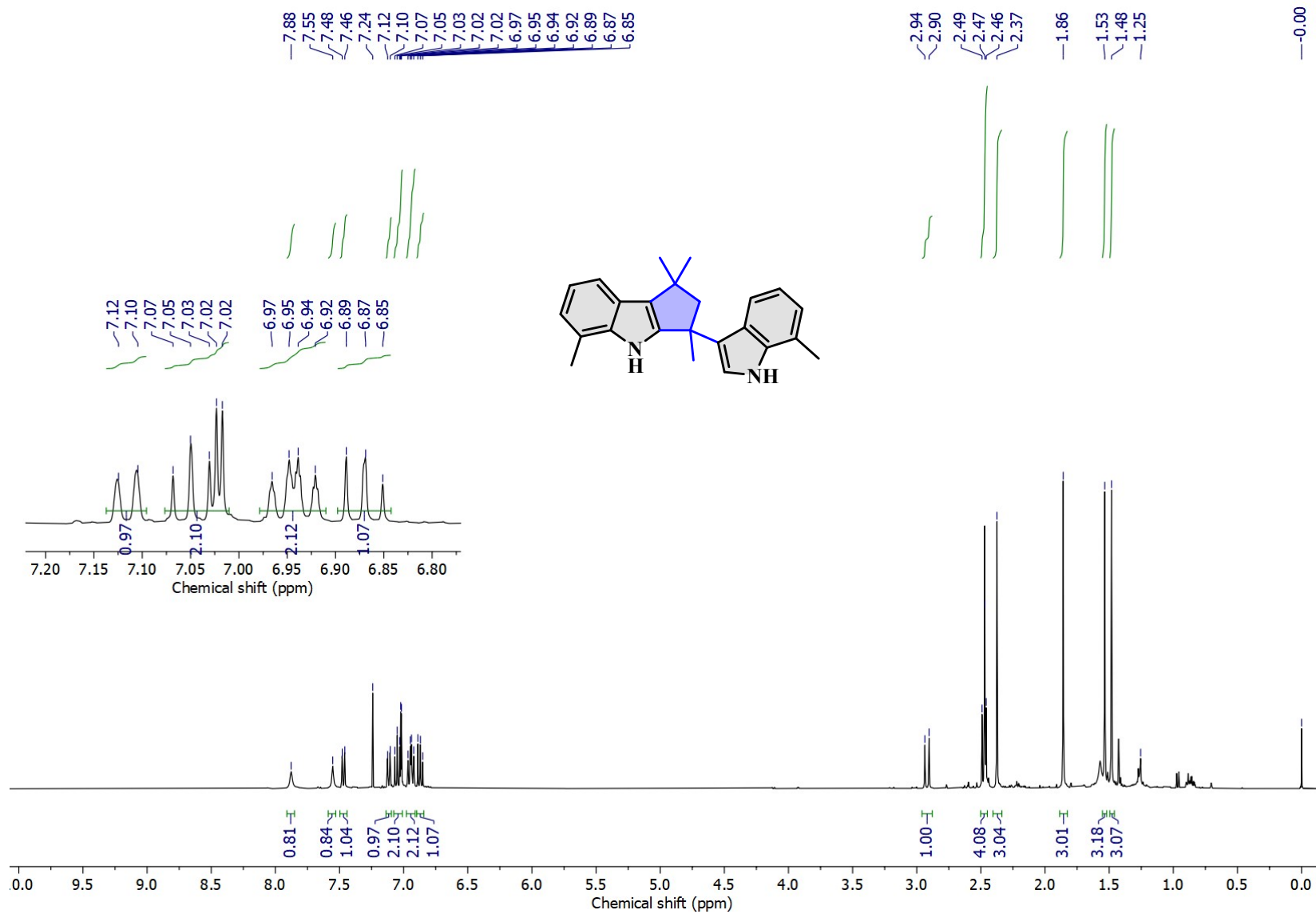


Fig. S13 400 MHz <sup>1</sup>H NMR spectrum of (2g)<sup>1</sup> in CDCl<sub>3</sub>

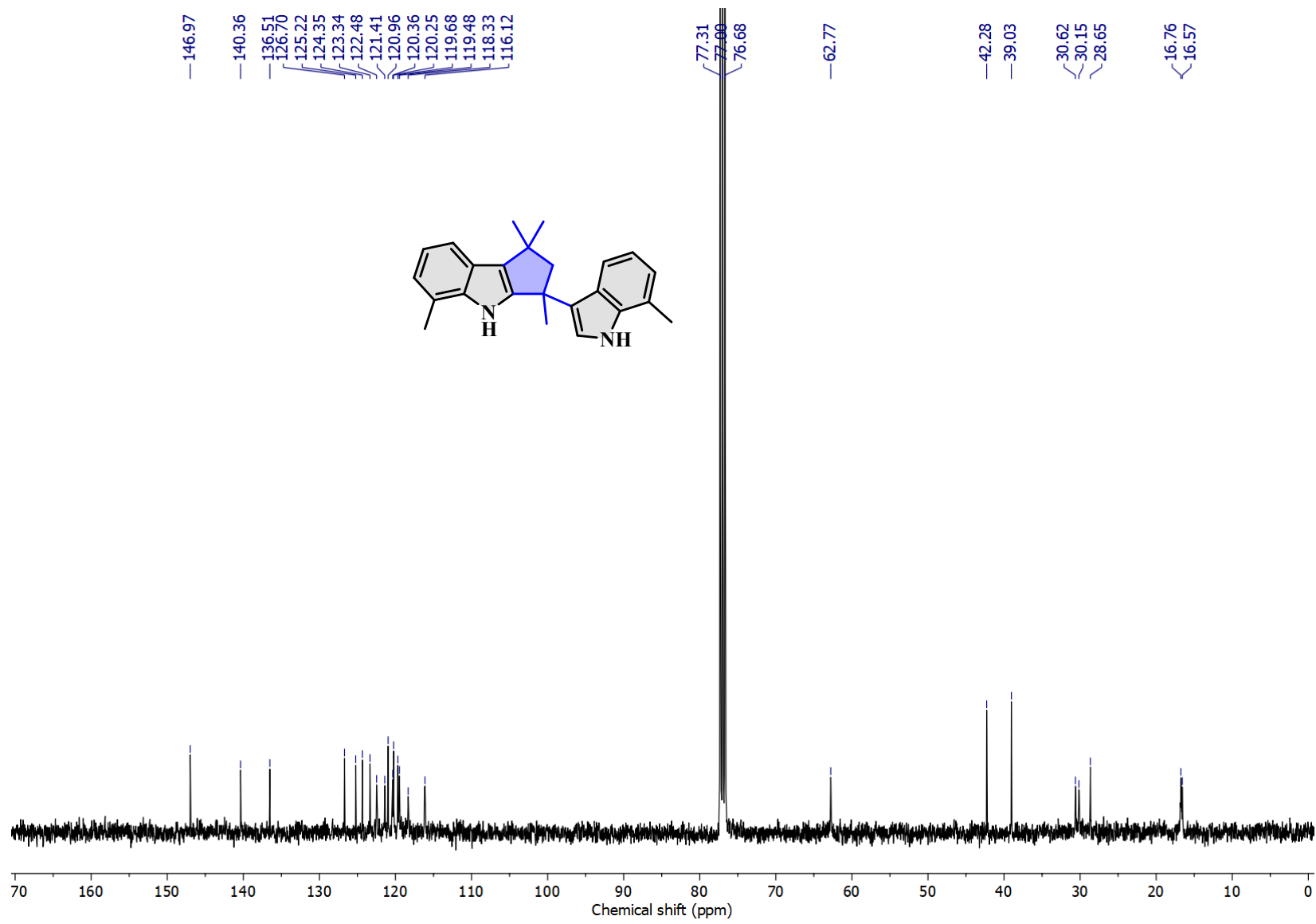
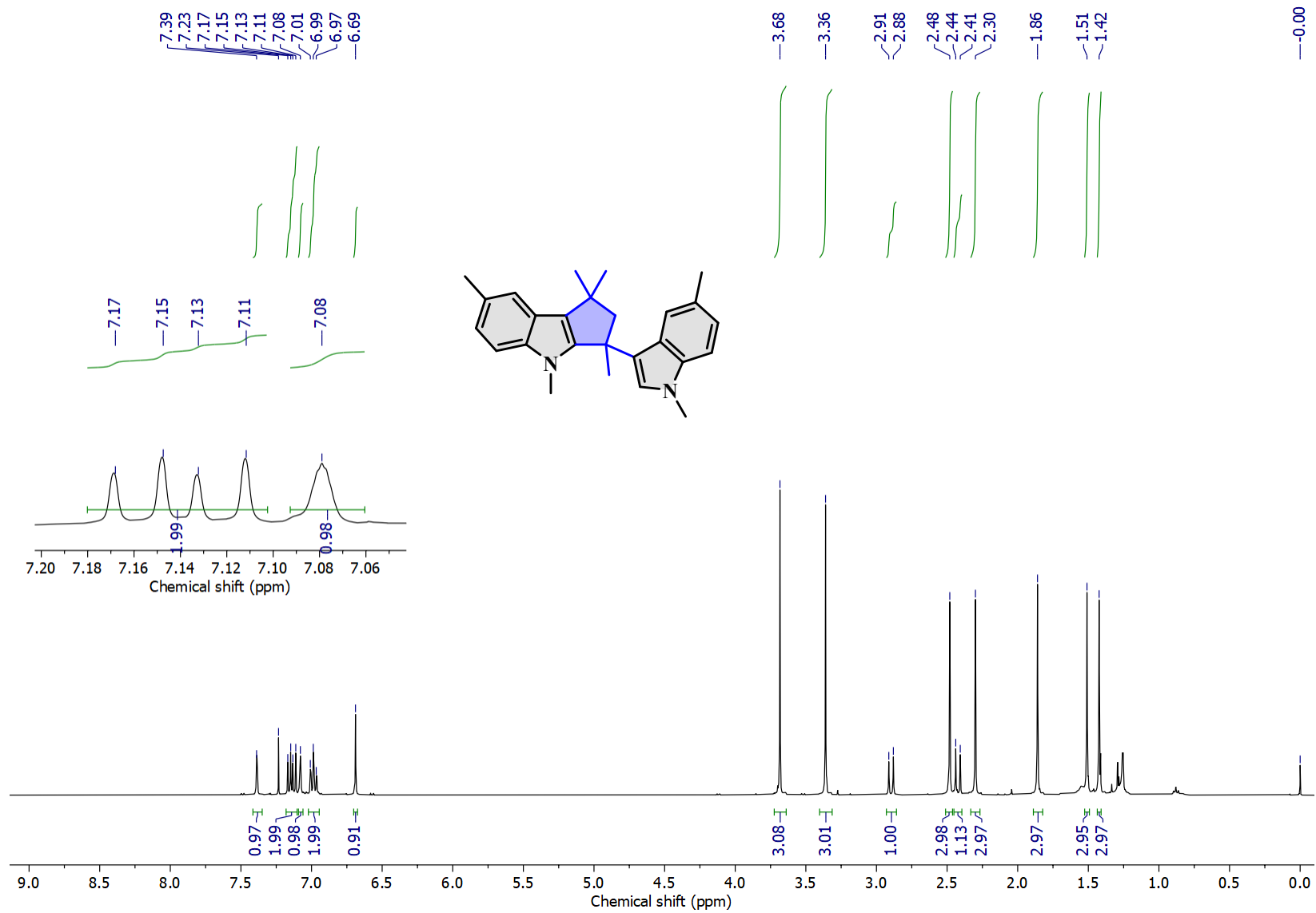


Fig. S14 100 MHz <sup>13</sup>C NMR spectrum of (2g)<sup>1</sup> in CDCl<sub>3</sub>



**Fig. S15** 400 MHz <sup>1</sup>H NMR spectrum of **(2h)<sup>2</sup>** in CDCl<sub>3</sub>



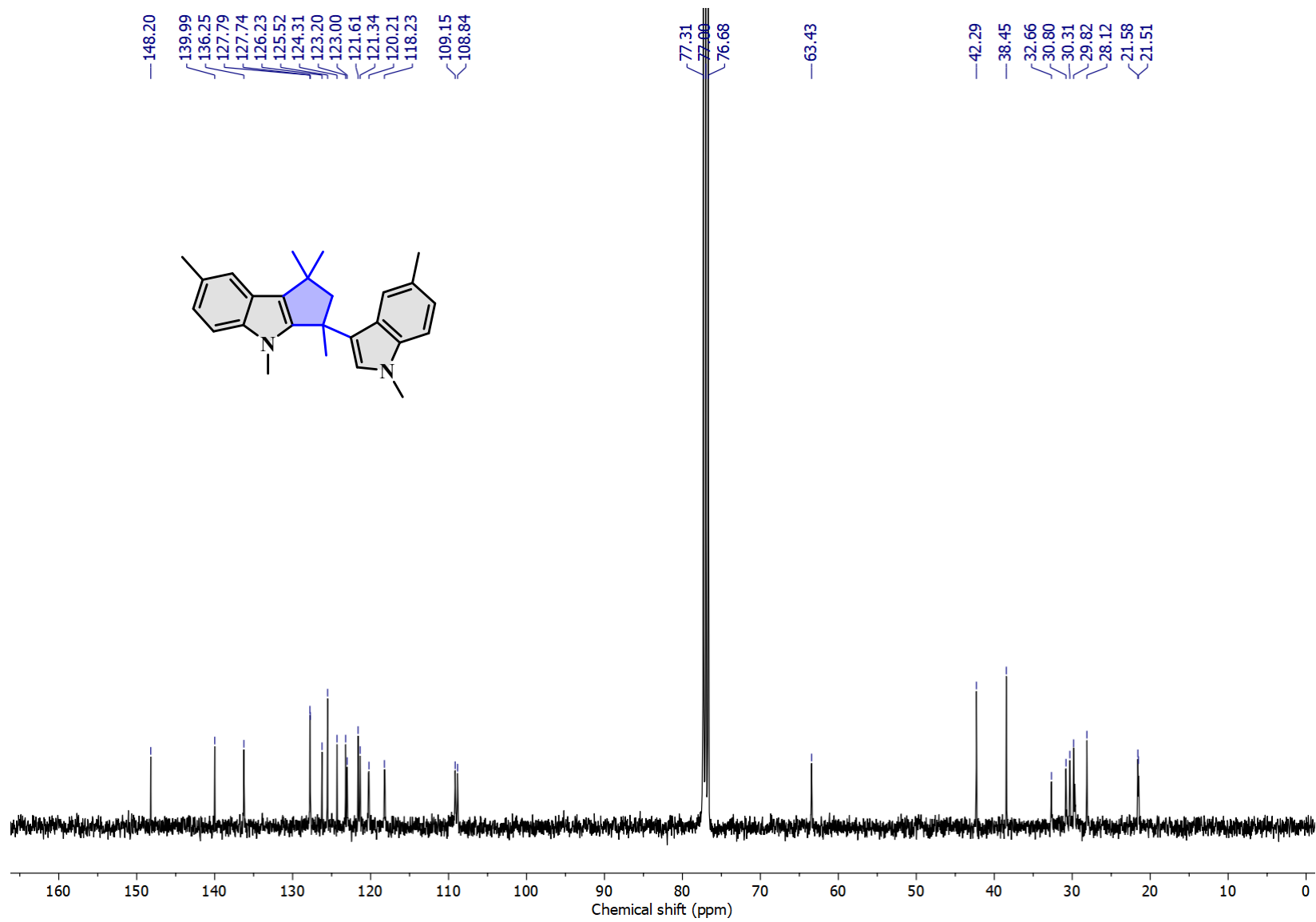
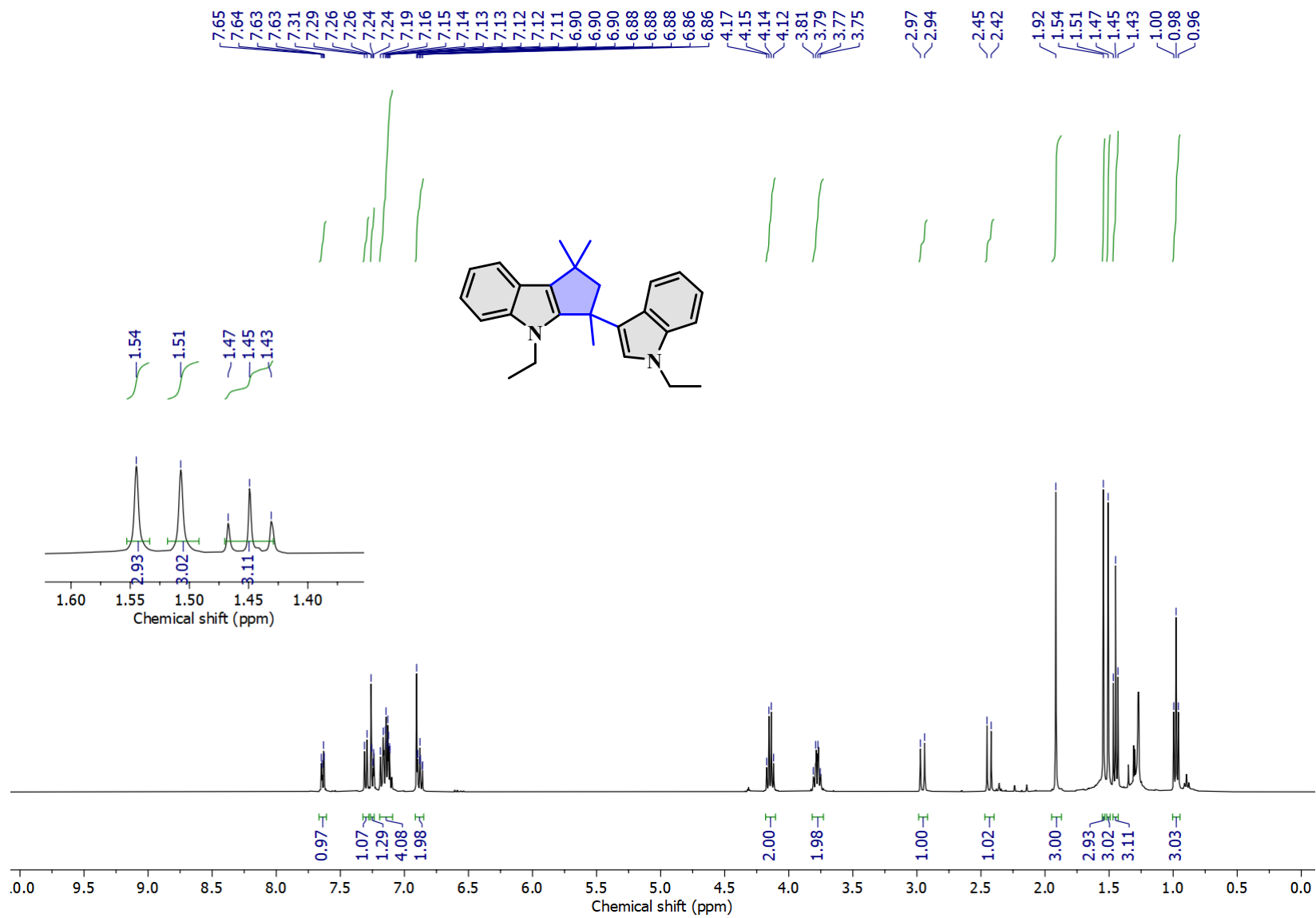


Fig. S16 100 MHz  $^{13}\text{C}$  NMR spectrum of  $(2h)_2$  in  $\text{CDCl}_3$



**Fig. S17** 400 MHz  $^1\text{H}$  NMR spectrum of **2i** in  $\text{CDCl}_3$

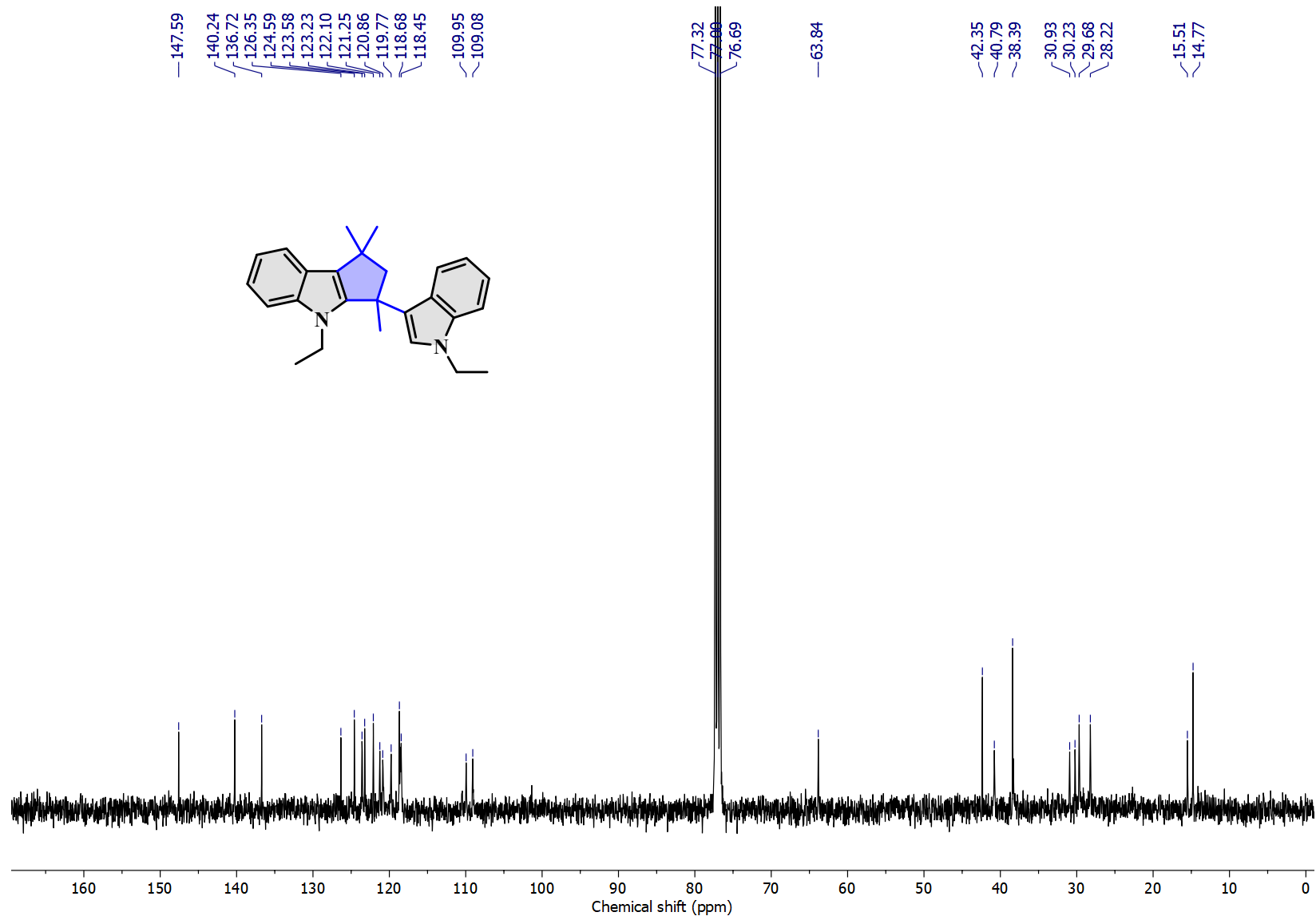


Fig. S18 100 MHz  $^{13}\text{C}$  NMR spectrum of **2i** in  $\text{CDCl}_3$

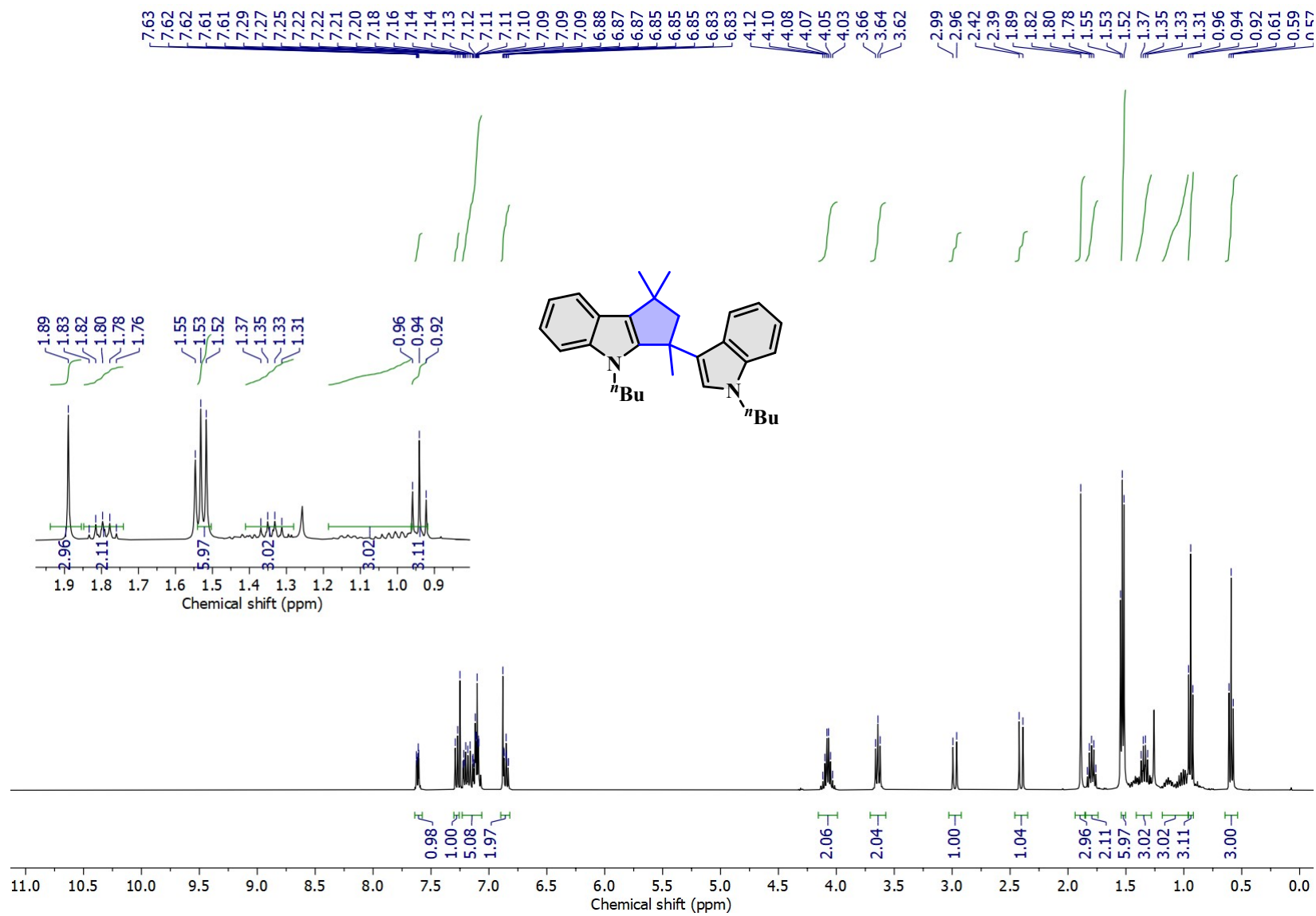


Fig. S19 400 MHz <sup>1</sup>H NMR spectrum of **2j** in CDCl<sub>3</sub>

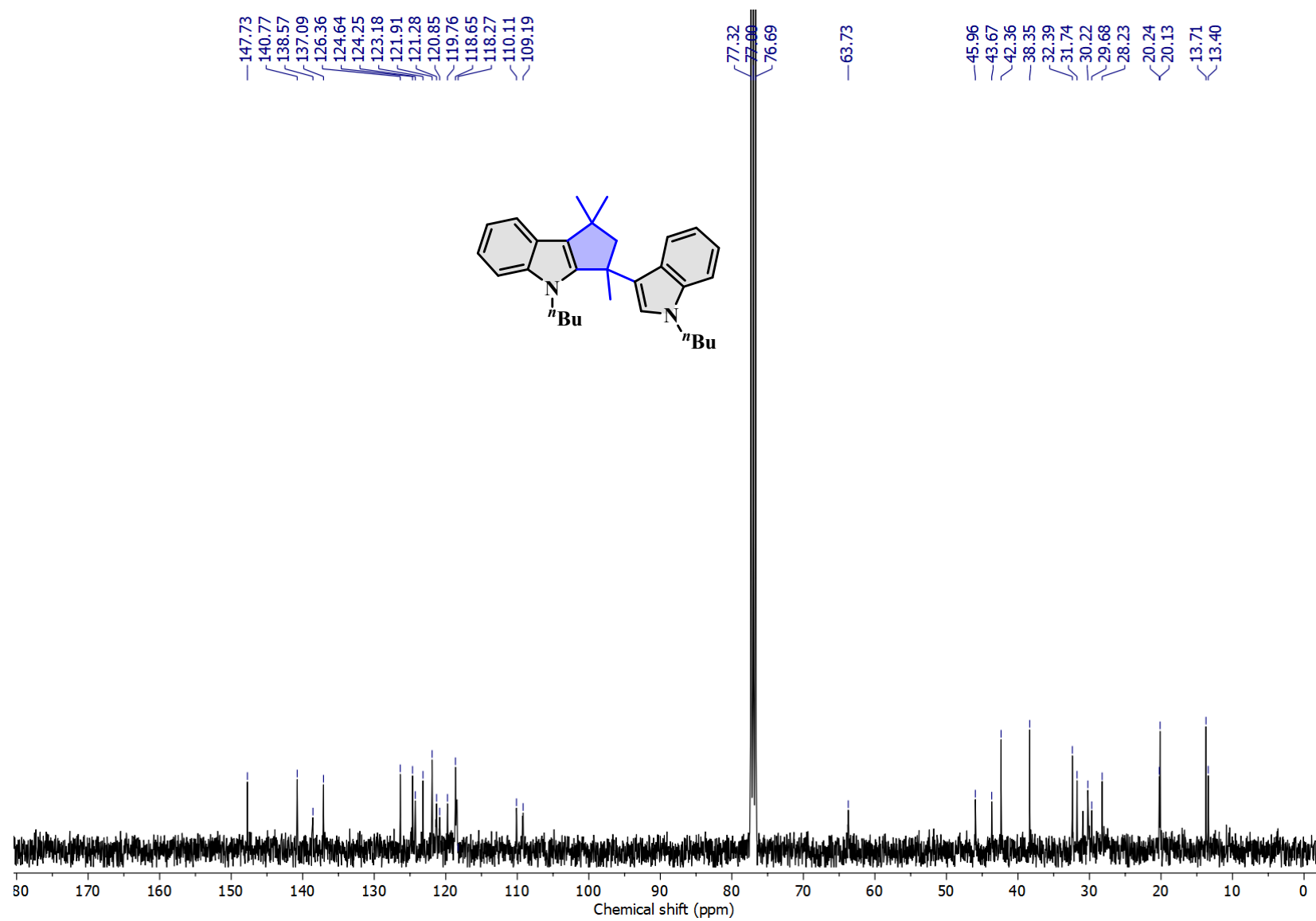


Fig. S20 100 MHz  $^{13}\text{C}$  NMR spectrum of **2j** in  $\text{CDCl}_3$

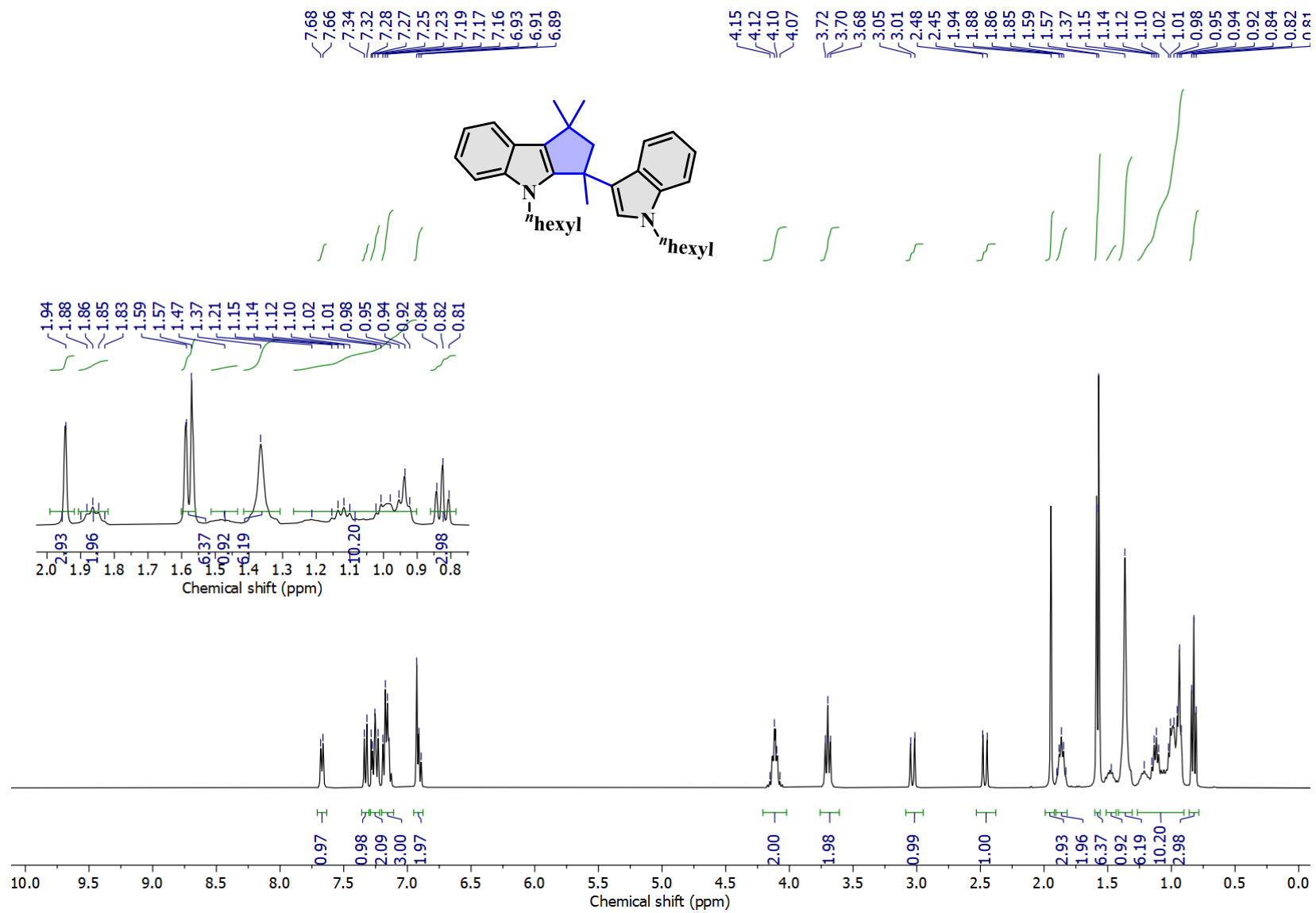
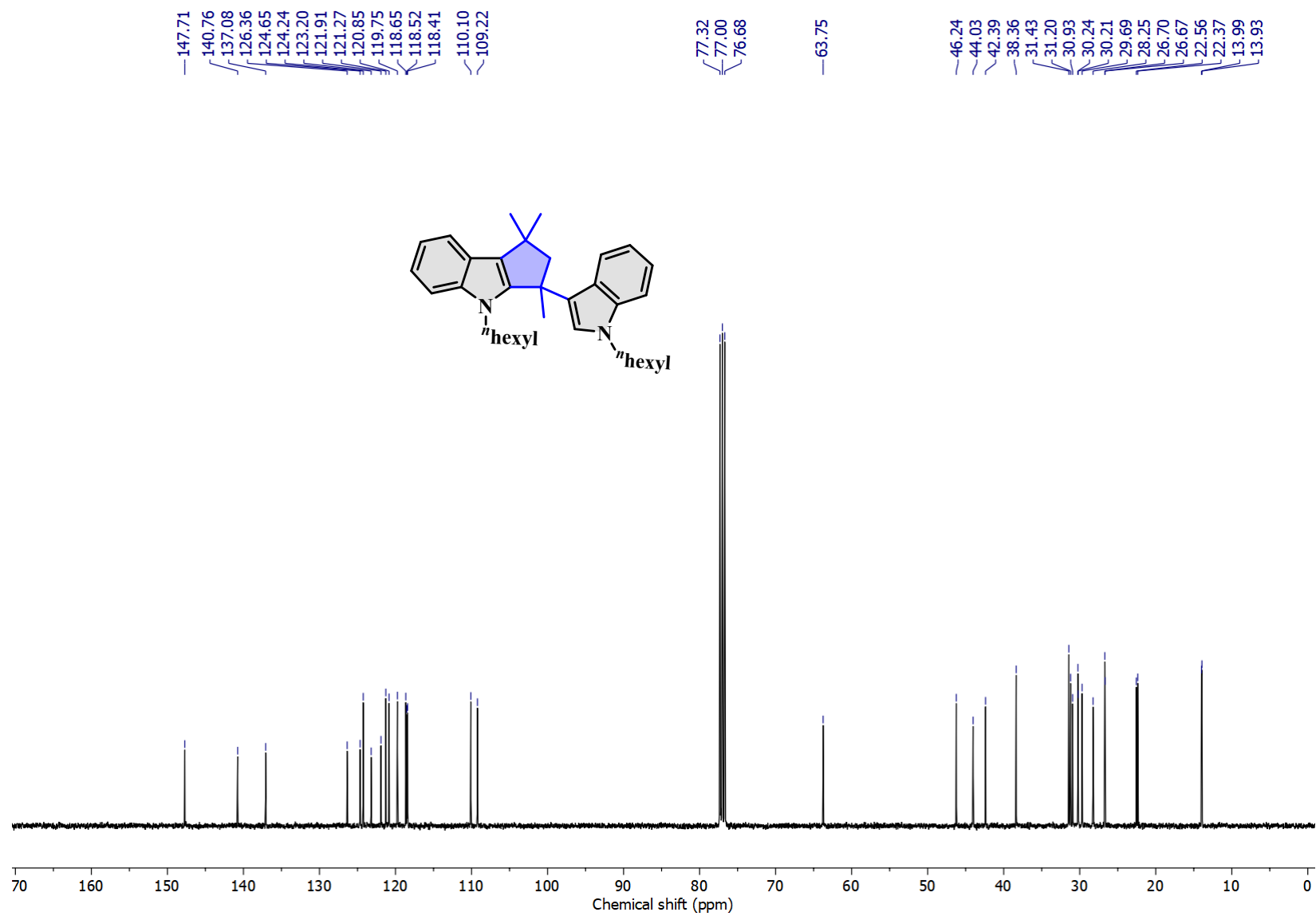


Fig. S21 400 MHz <sup>1</sup>H NMR spectrum of **2k** in CDCl<sub>3</sub>



**Fig. S22** 100 MHz  $^{13}\text{C}$  NMR spectrum of **2k** in  $\text{CDCl}_3$

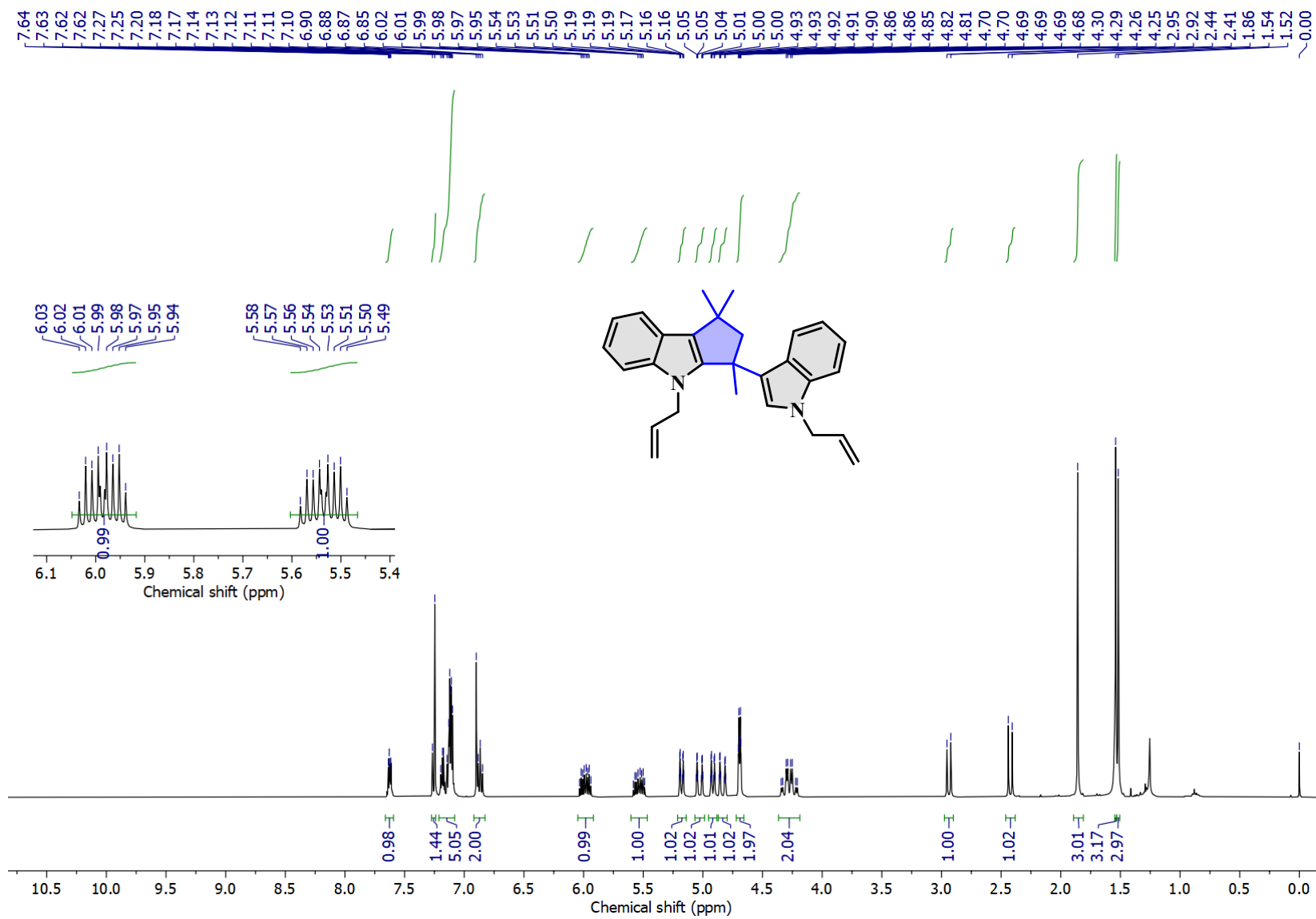


Fig. S23 400 MHz  $^1\text{H}$  NMR spectrum of **21** in  $\text{CDCl}_3$



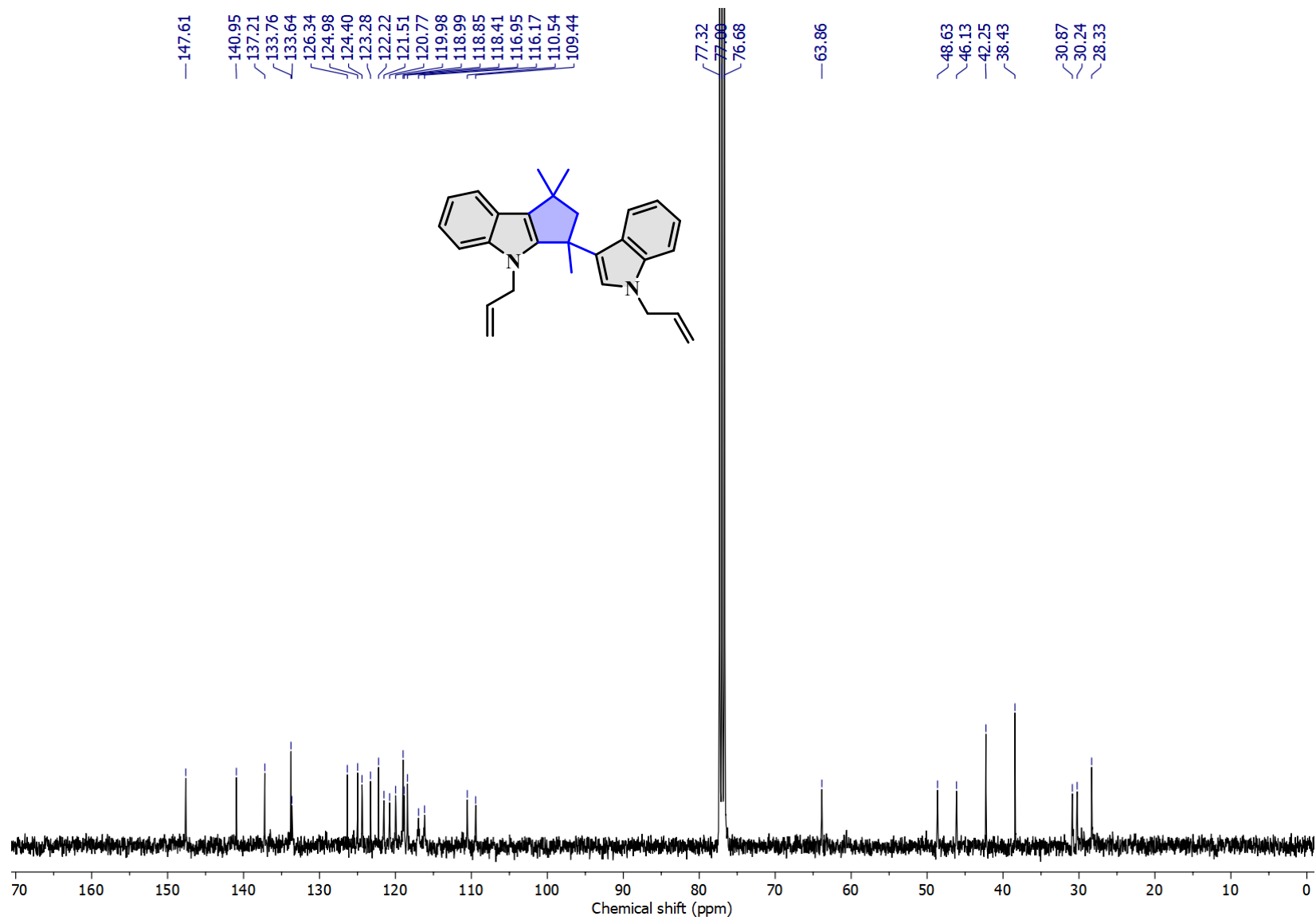


Fig. S24 100 MHz <sup>13</sup>C NMR spectrum of **2I** in CDCl<sub>3</sub>

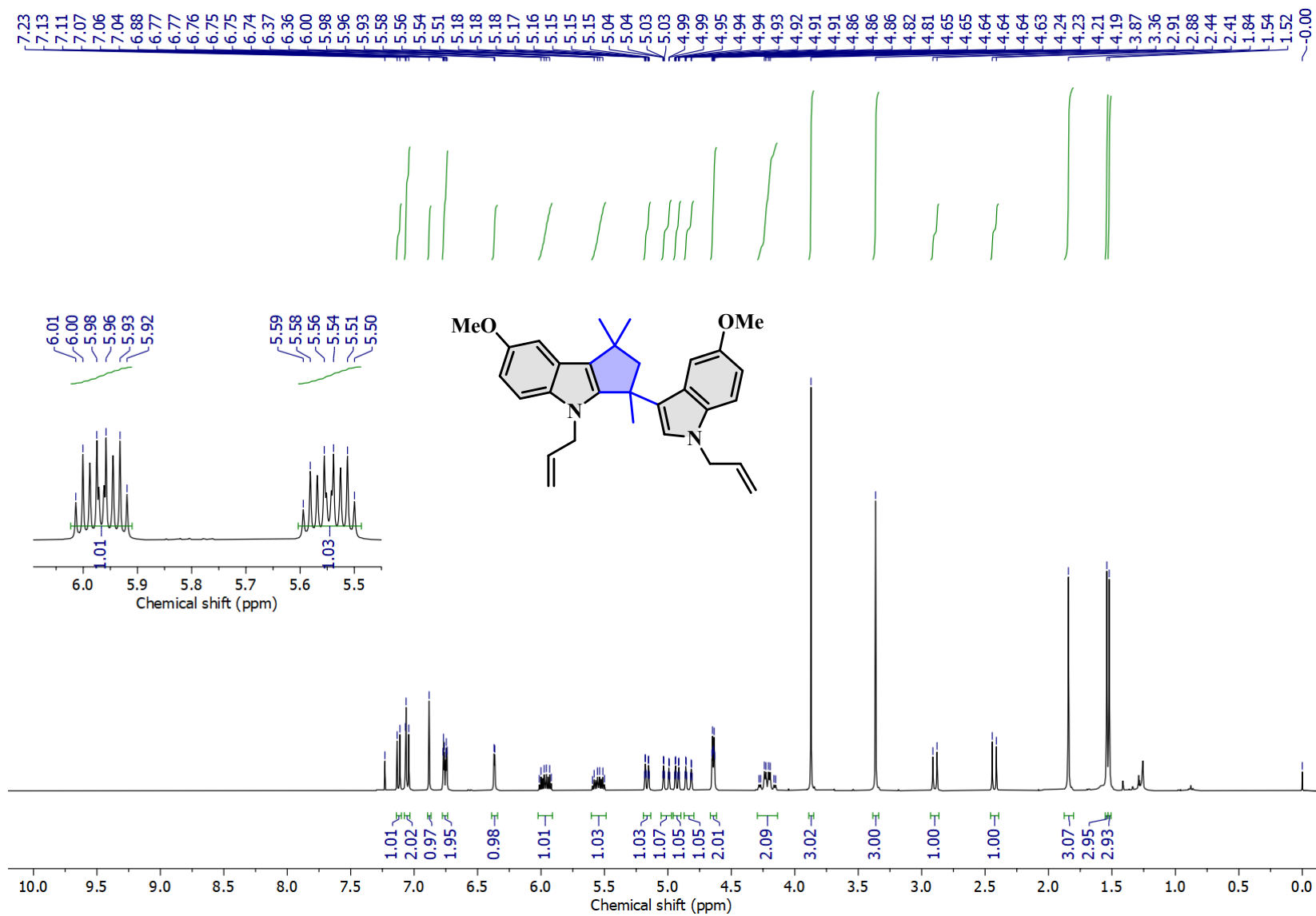


Fig. S25 400 MHz  $^1\text{H}$  NMR spectrum of **2m** in  $\text{CDCl}_3$

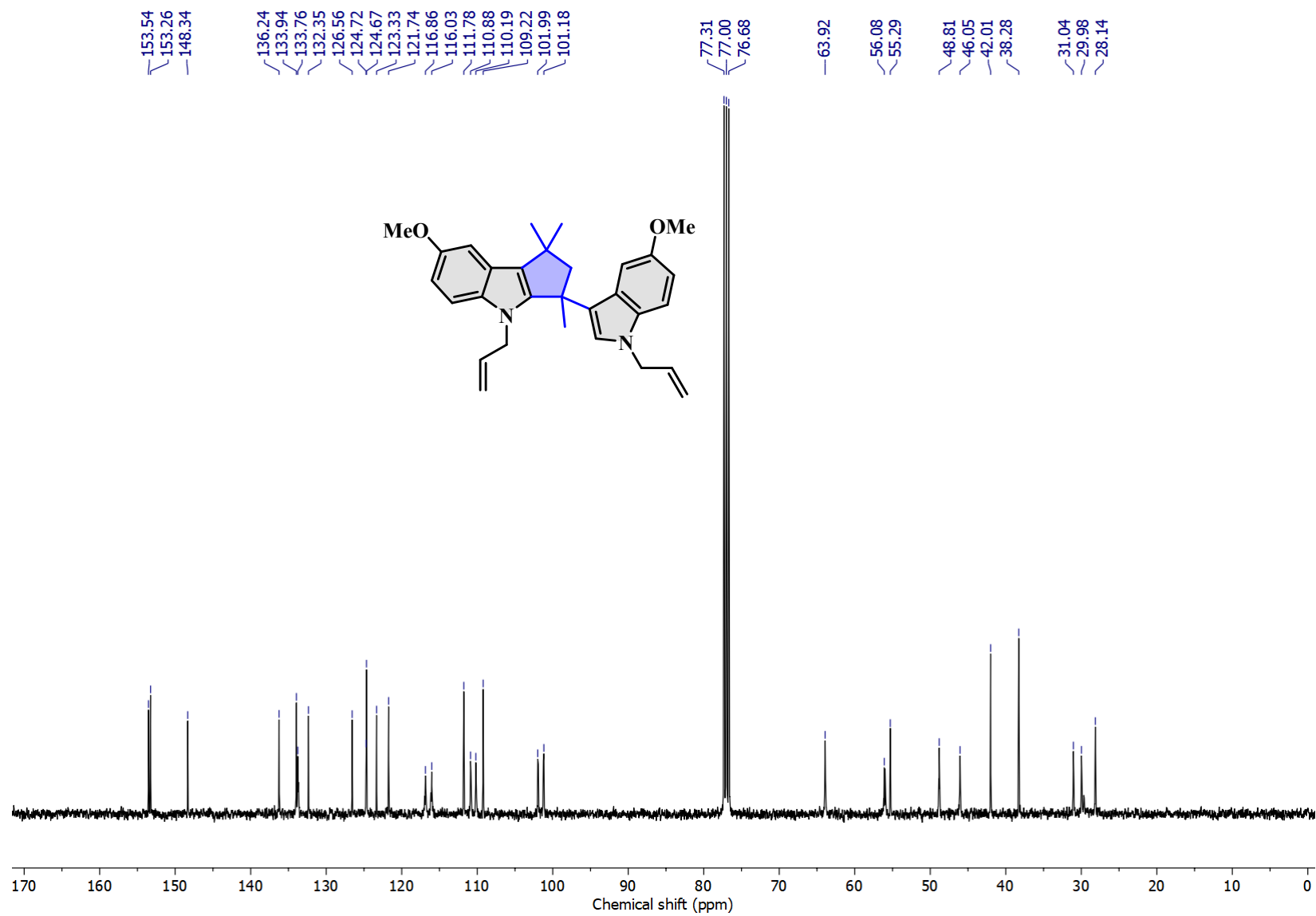


Fig. S26 100 MHz  $^{13}\text{C}$  NMR spectrum of **2m** in  $\text{CDCl}_3$

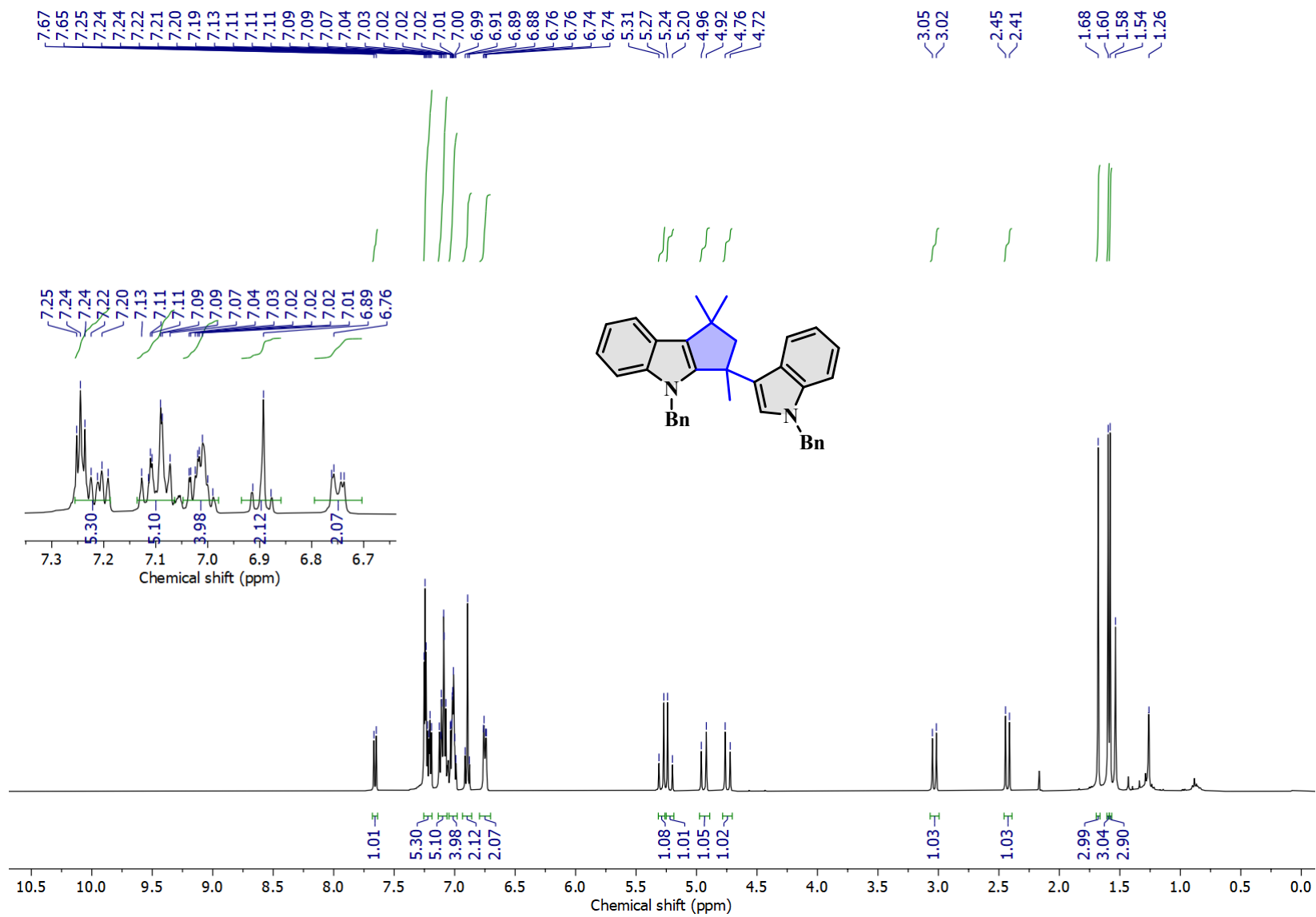


Fig. S27 400 MHz <sup>1</sup>H NMR spectrum of **2n** in CDCl<sub>3</sub>

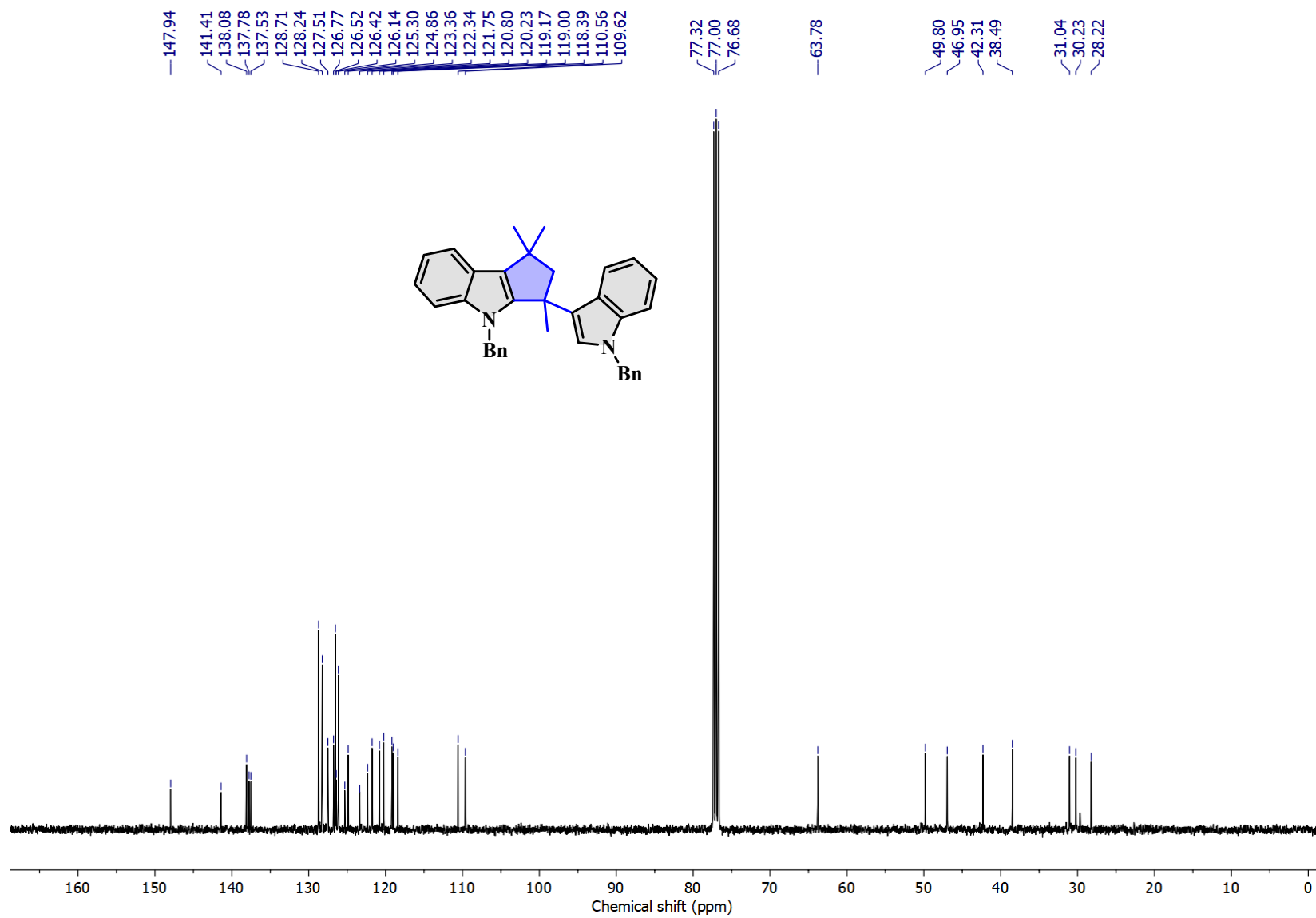


Fig. S28 100 MHz  $^{13}\text{C}$  NMR spectrum of **2n** in  $\text{CDCl}_3$

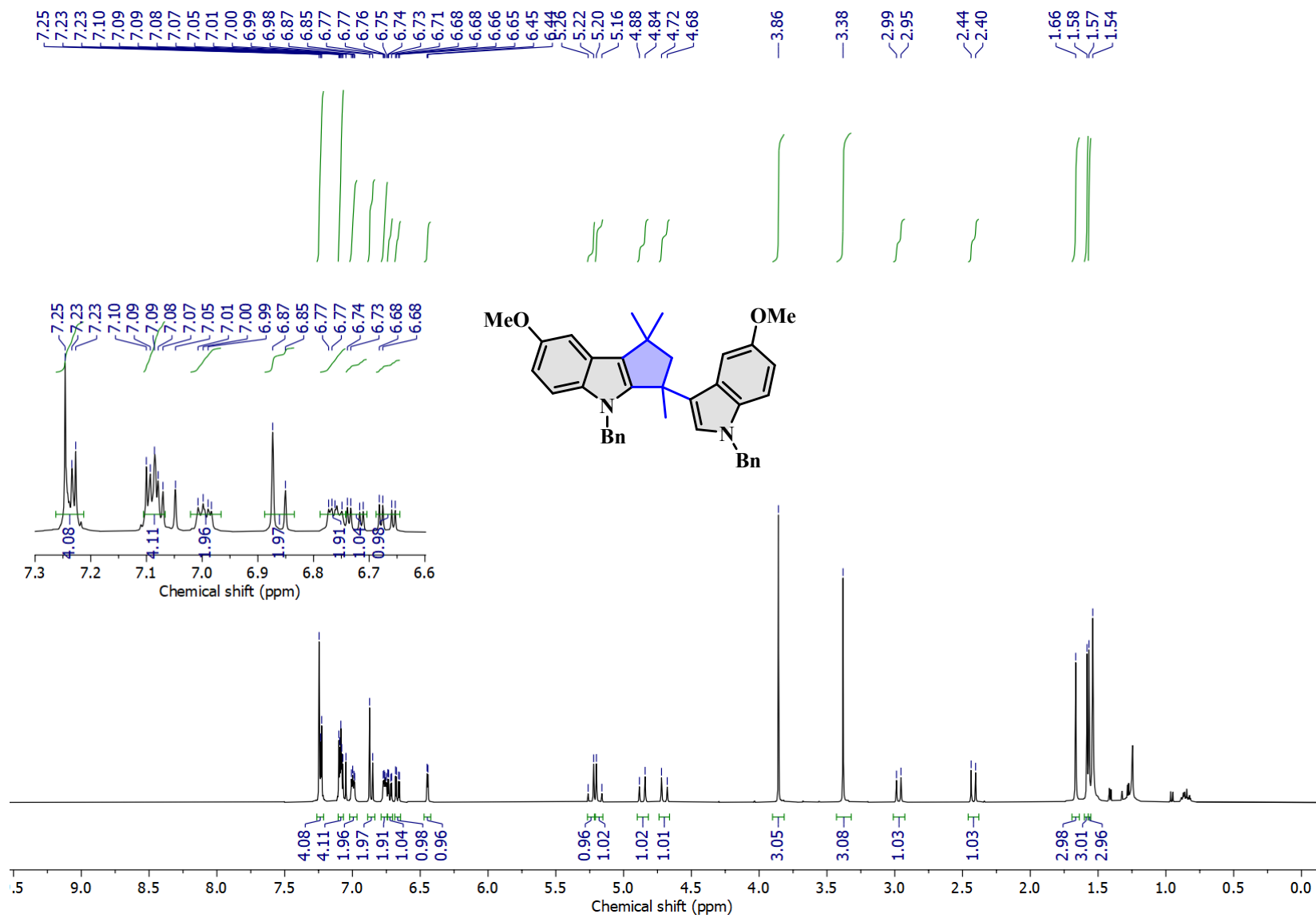


Fig. S29 400 MHz  $^1\text{H}$  NMR spectrum of **2o** in  $\text{CDCl}_3$

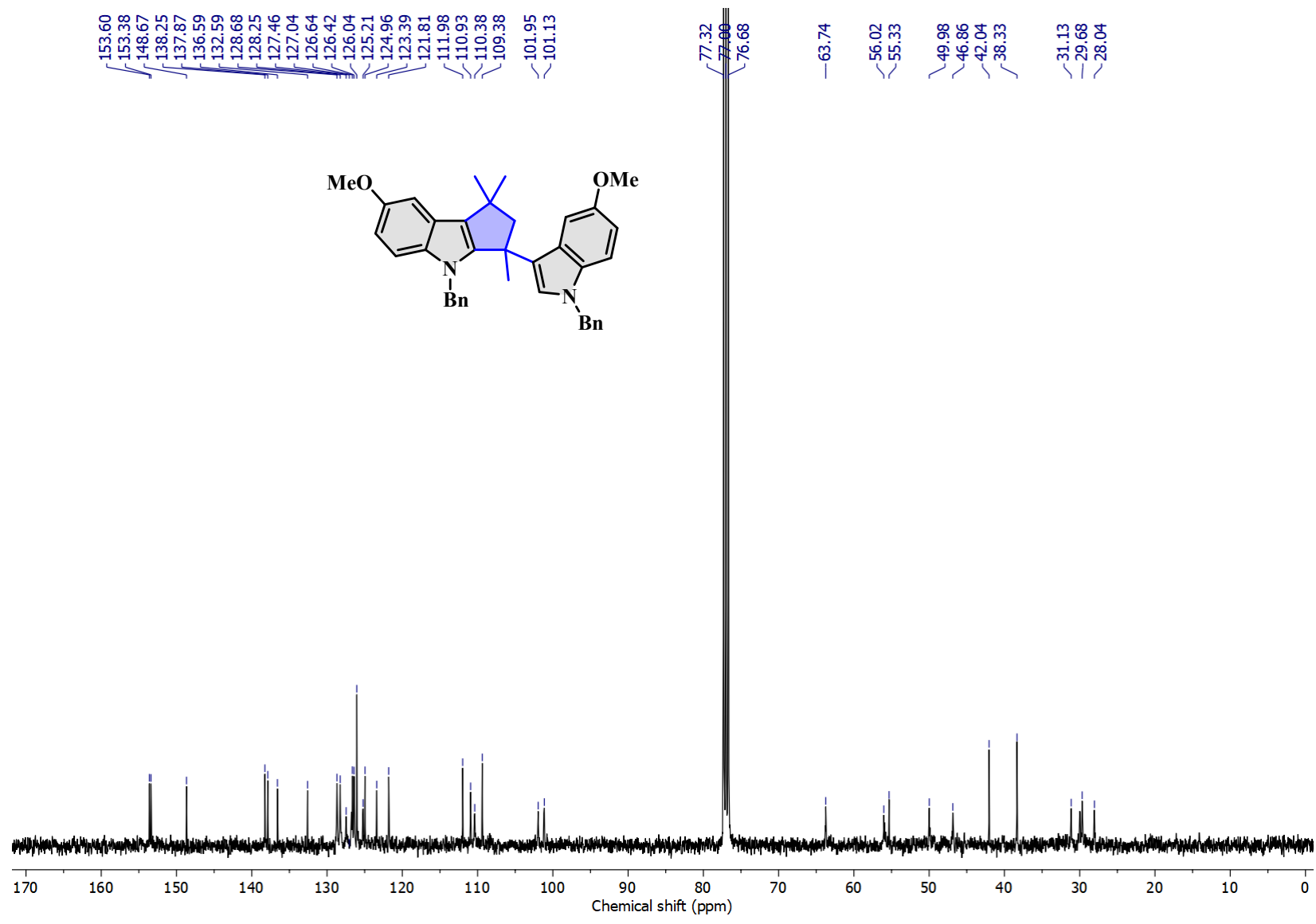


Fig. S30 100 MHz  $^{13}\text{C}$  NMR spectrum of **2o** in  $\text{CDCl}_3$

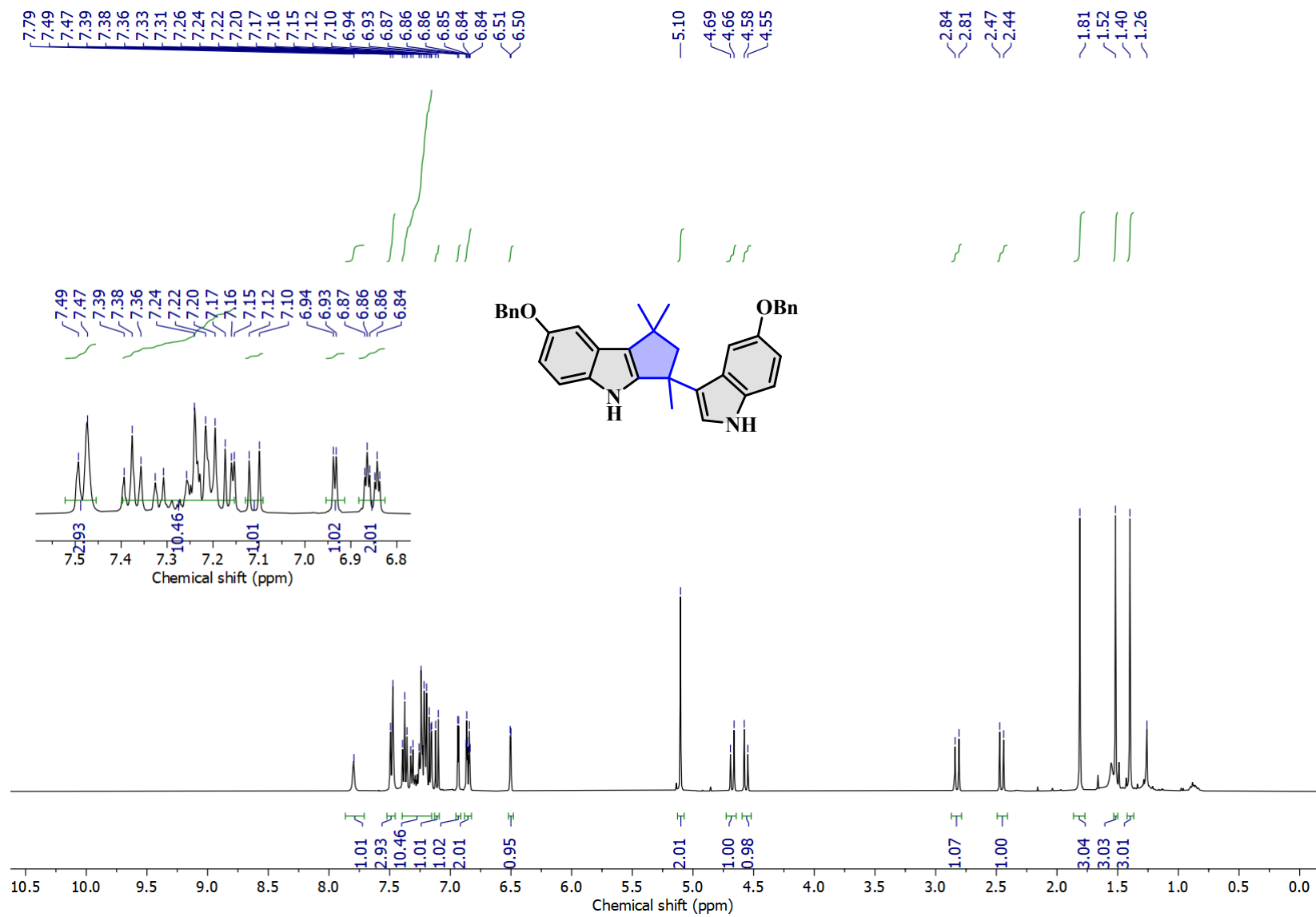


Fig. S31 400 MHz <sup>1</sup>H NMR spectrum of **2p** in CDCl<sub>3</sub>



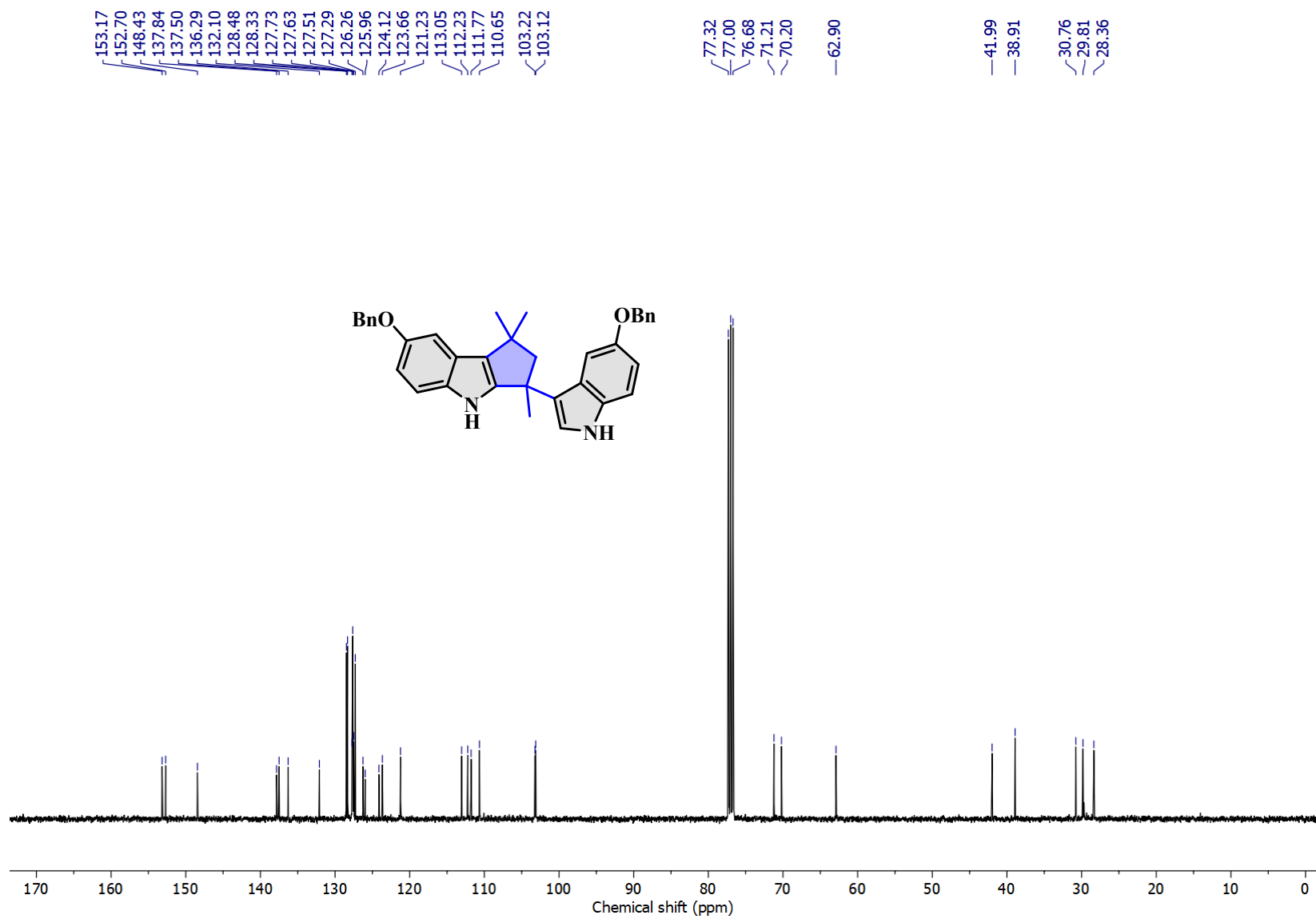


Fig. S32 100 MHz  $^{13}\text{C}$  NMR spectrum of **2p** in  $\text{CDCl}_3$

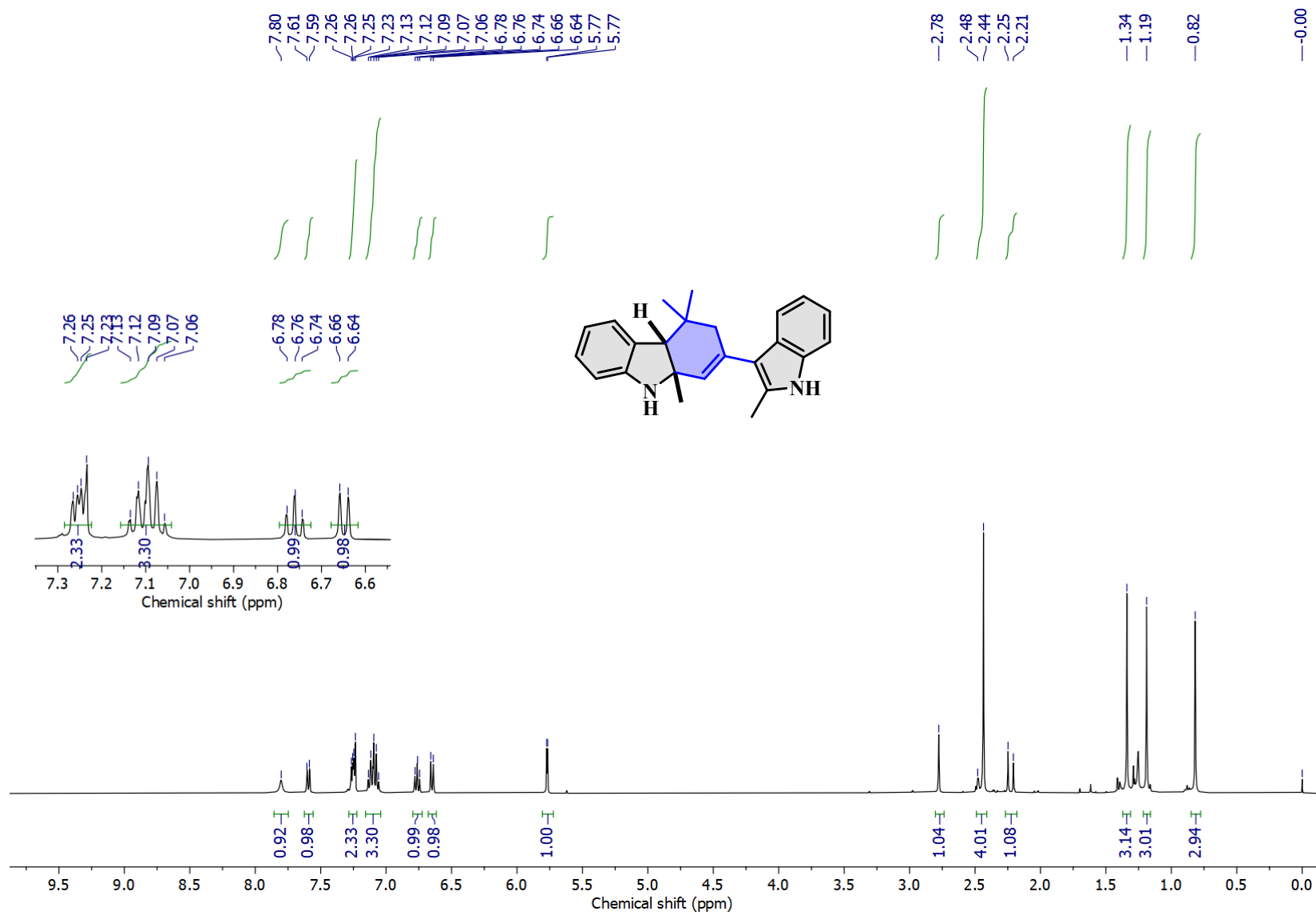


Fig. S33 400 MHz <sup>1</sup>H NMR spectrum of (2q)<sup>3</sup> in CDCl<sub>3</sub>

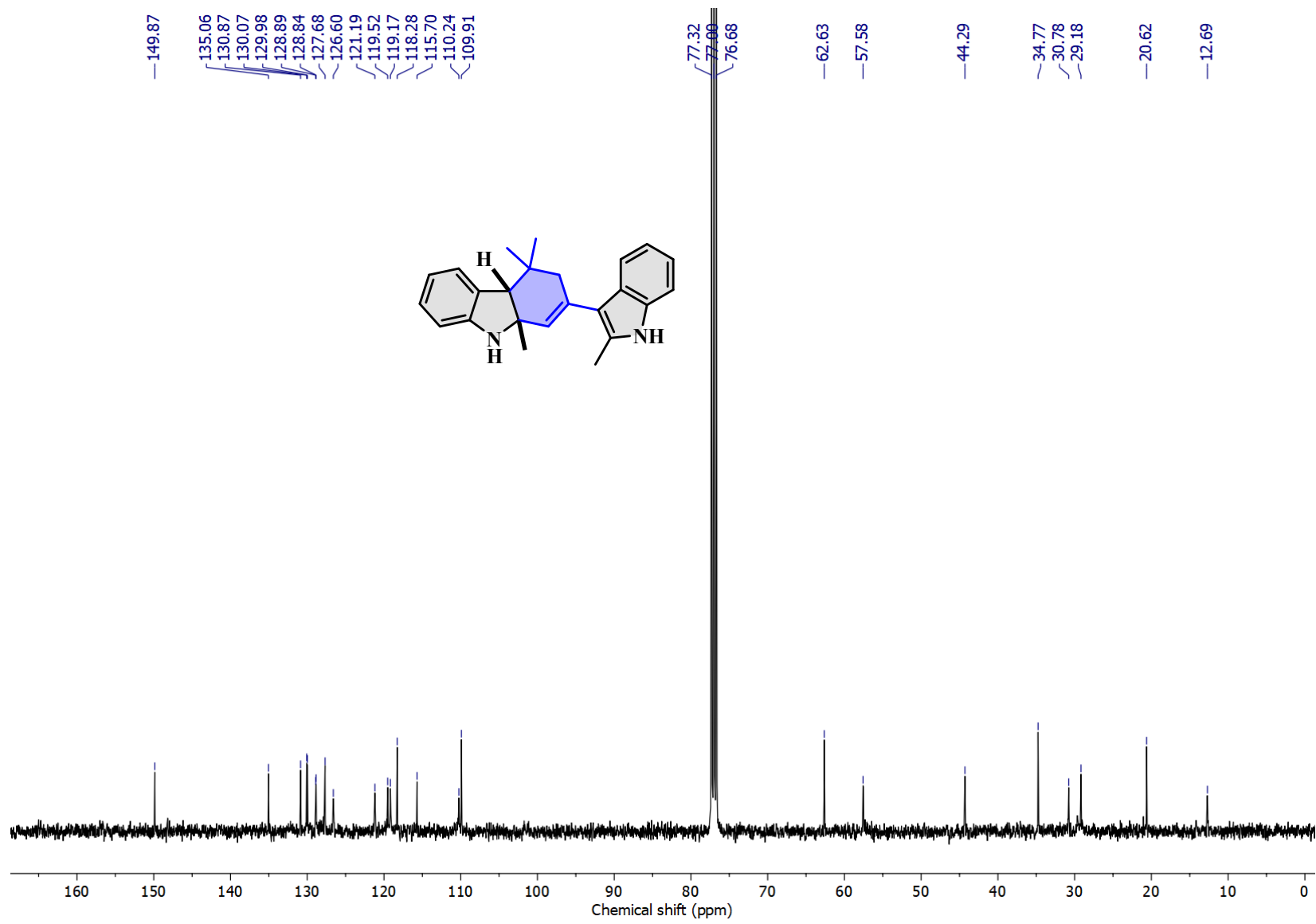
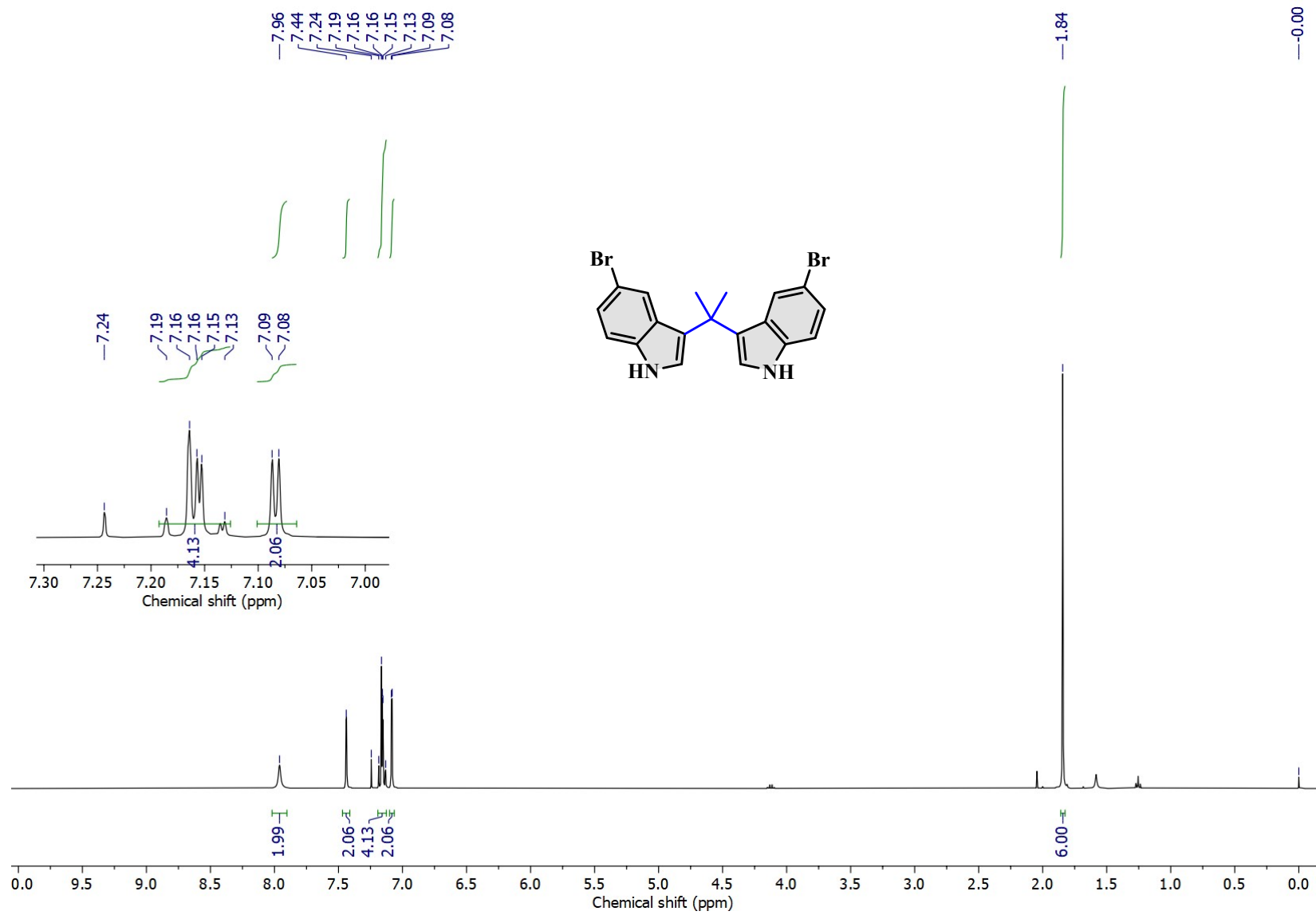


Fig. S34 100 MHz <sup>13</sup>C NMR spectrum of (2q)<sup>3</sup> in CDCl<sub>3</sub>



**Fig. S35** 400 MHz  $^1\text{H NMR}$  spectrum of  $(\mathbf{3a})_2$  in  $\text{CDCl}_3$

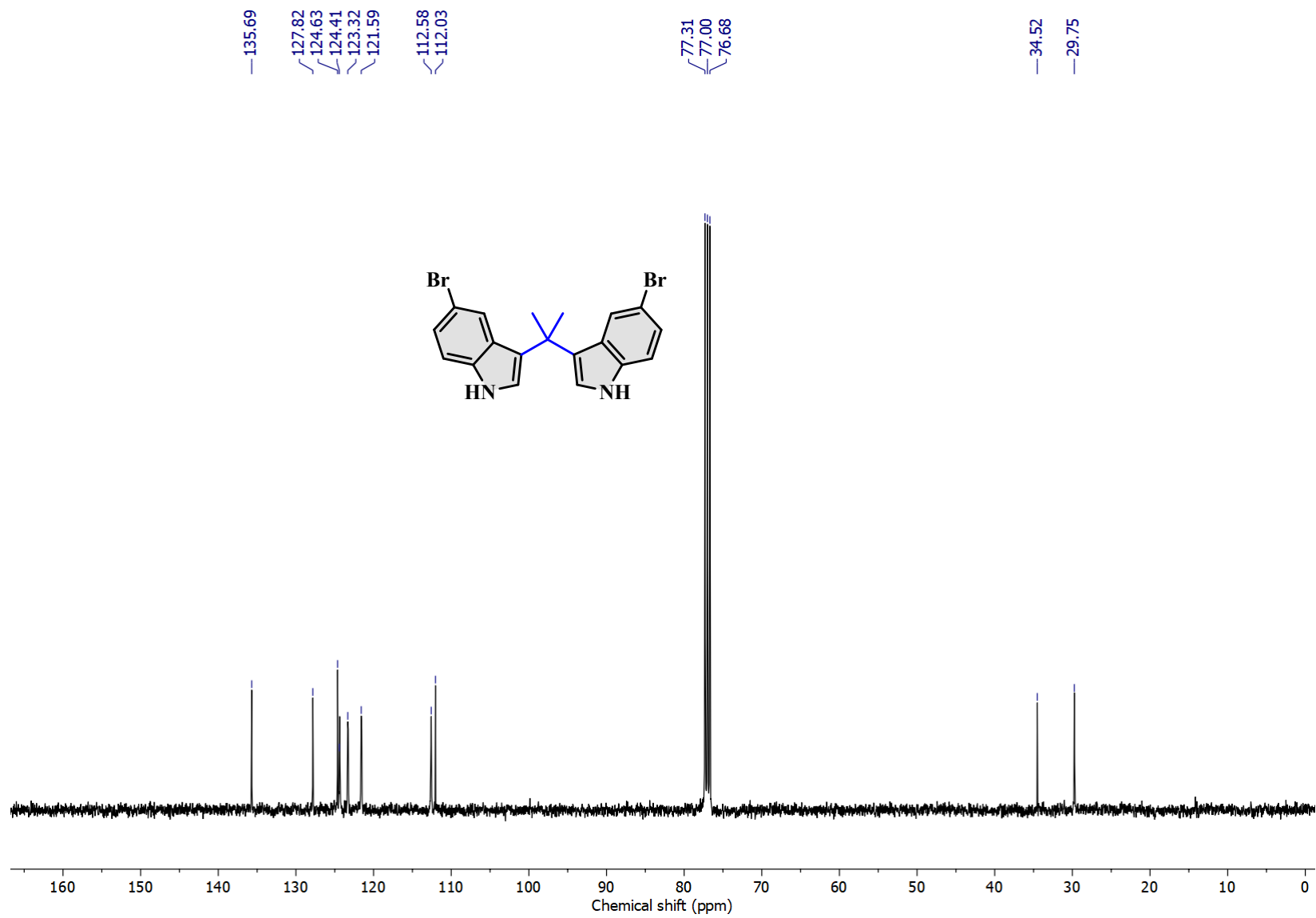


Fig. S36 100 MHz <sup>13</sup>C NMR spectrum of (3a)<sub>2</sub> in CDCl<sub>3</sub>

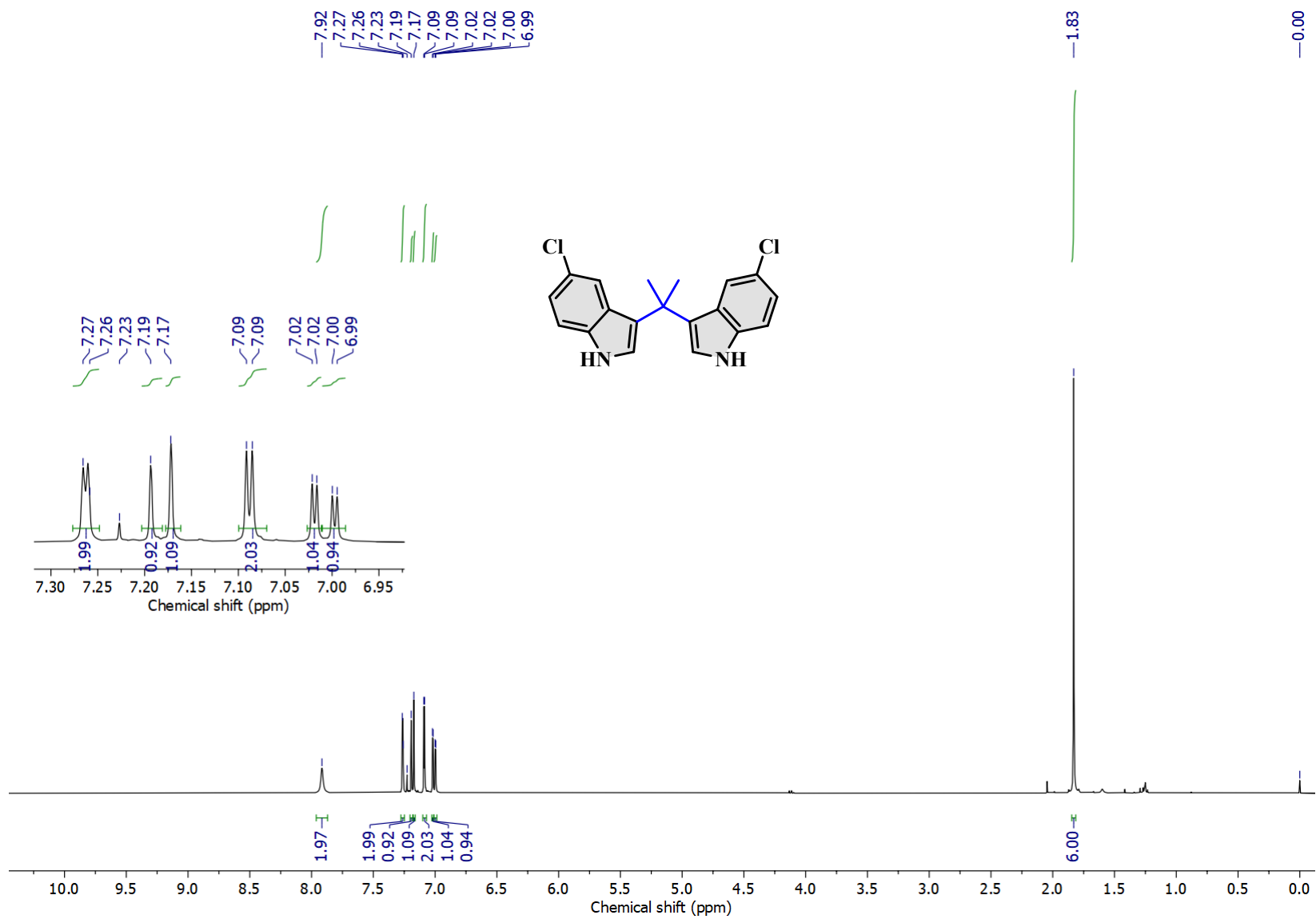


Fig. S37 400 MHz <sup>1</sup>H NMR spectrum of (3b)<sub>2</sub> in CDCl<sub>3</sub>

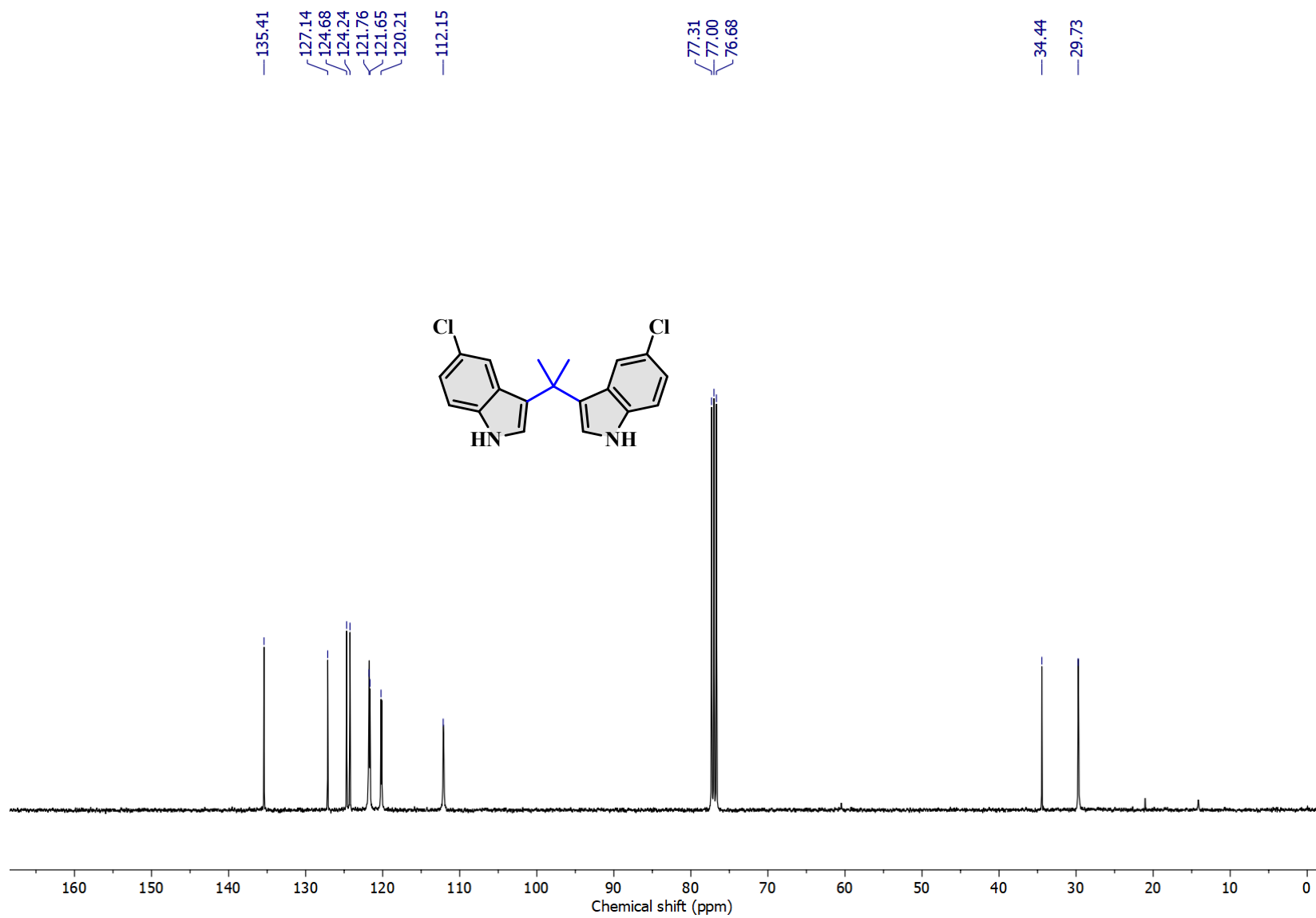
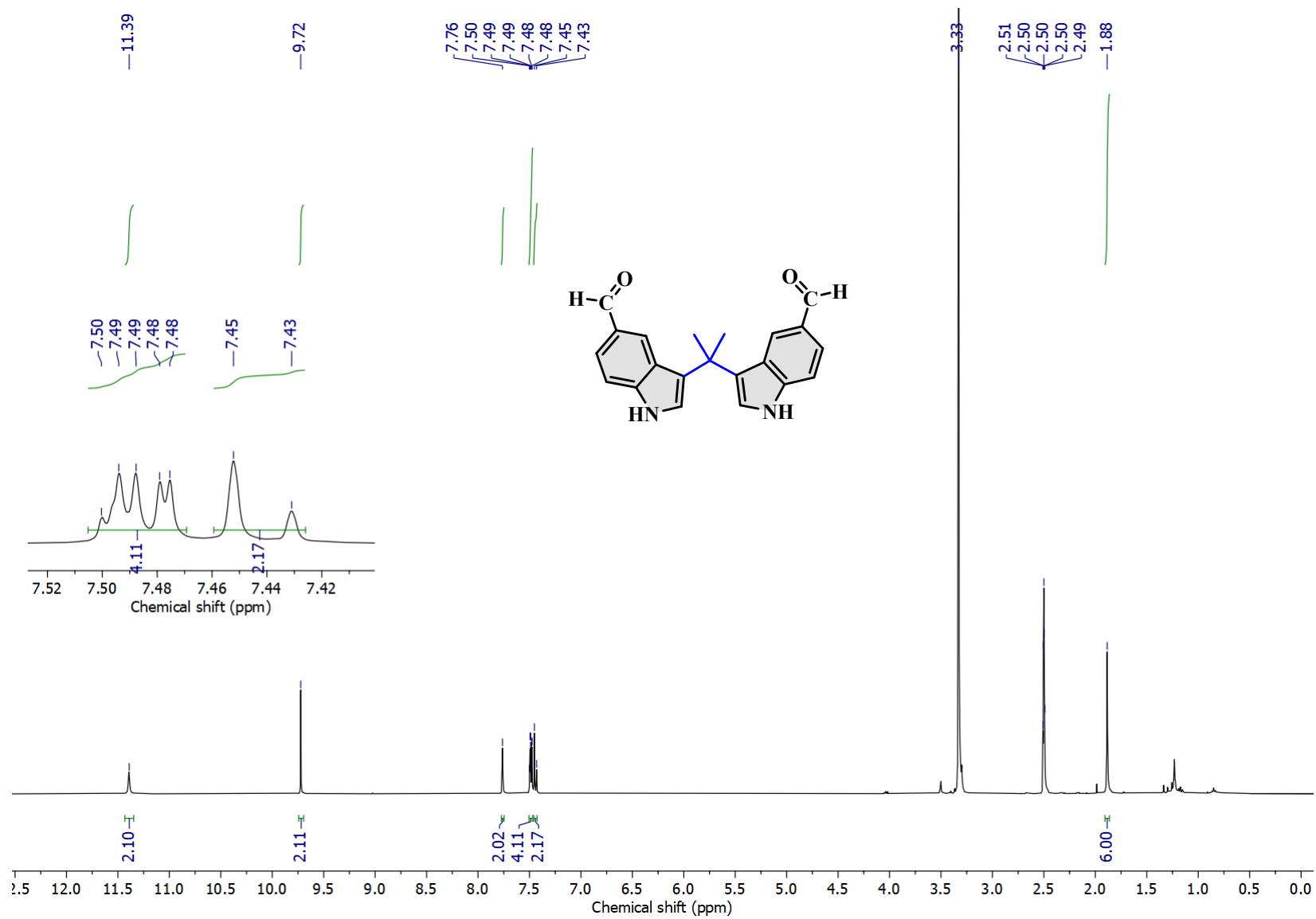


Fig. S38 100 MHz <sup>13</sup>C NMR spectrum of (3b)<sub>2</sub> in CDCl<sub>3</sub>



**Fig. S39** 400 MHz  $^1\text{H}$  NMR spectrum of **3c** in  $\text{DMSO-}d_6$



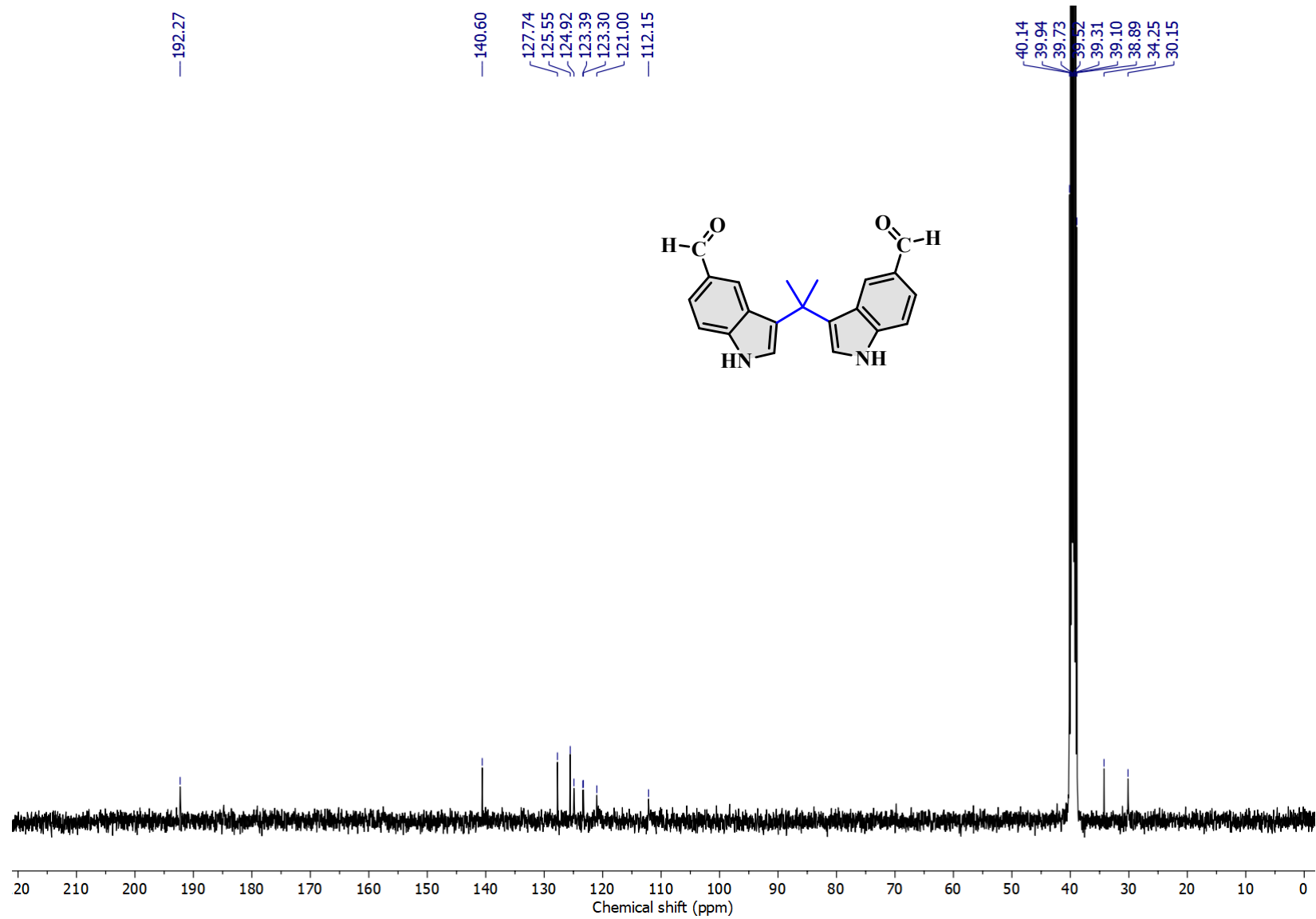
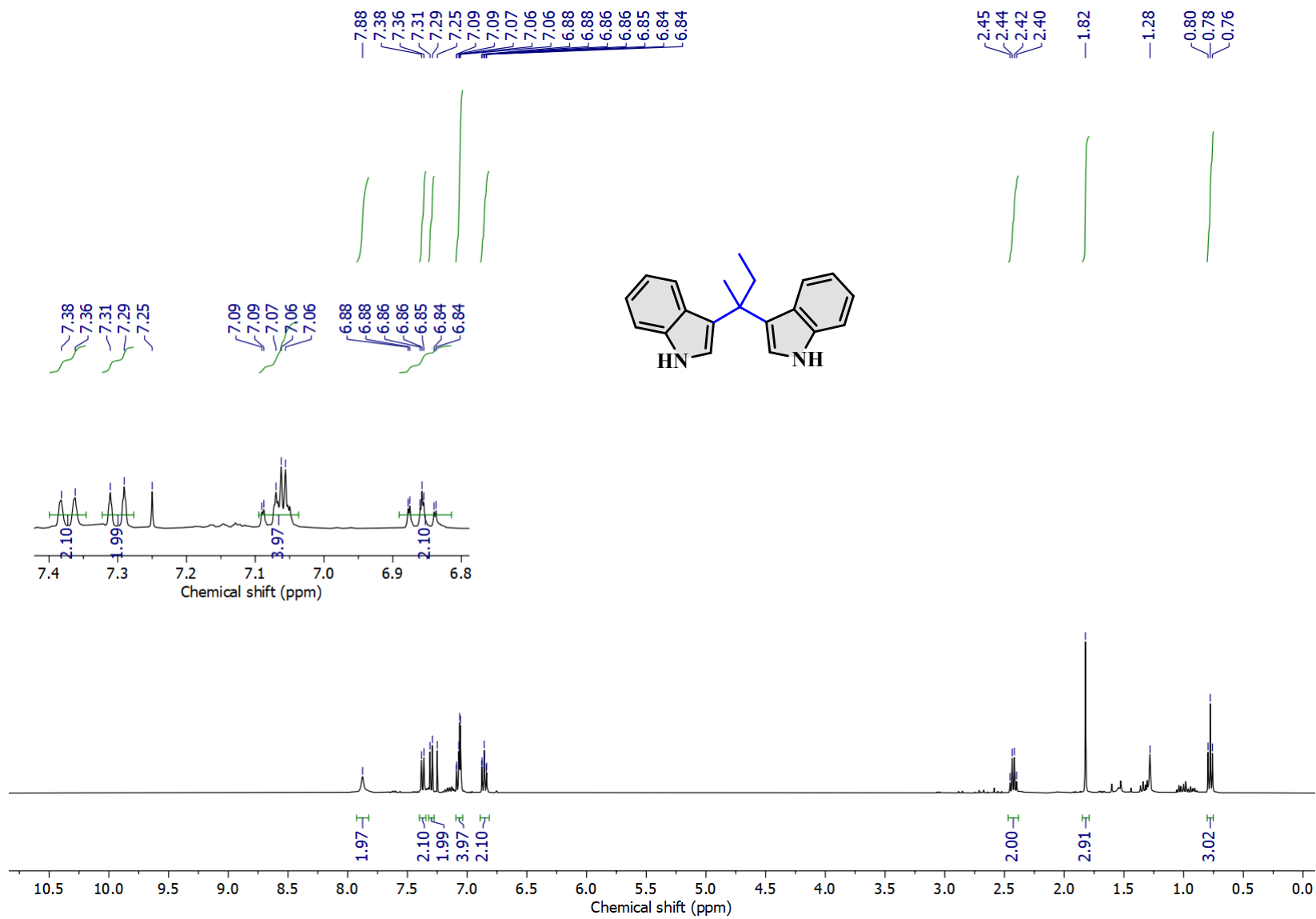


Fig. S40 100 MHz  $^{13}\text{C}$  NMR spectrum of **3c** in  $\text{DMSO-}d_6$



**Fig. S41** 400 MHz  $^1\text{H}$  NMR spectrum of **(3d)**<sup>4</sup> in  $\text{CDCl}_3$

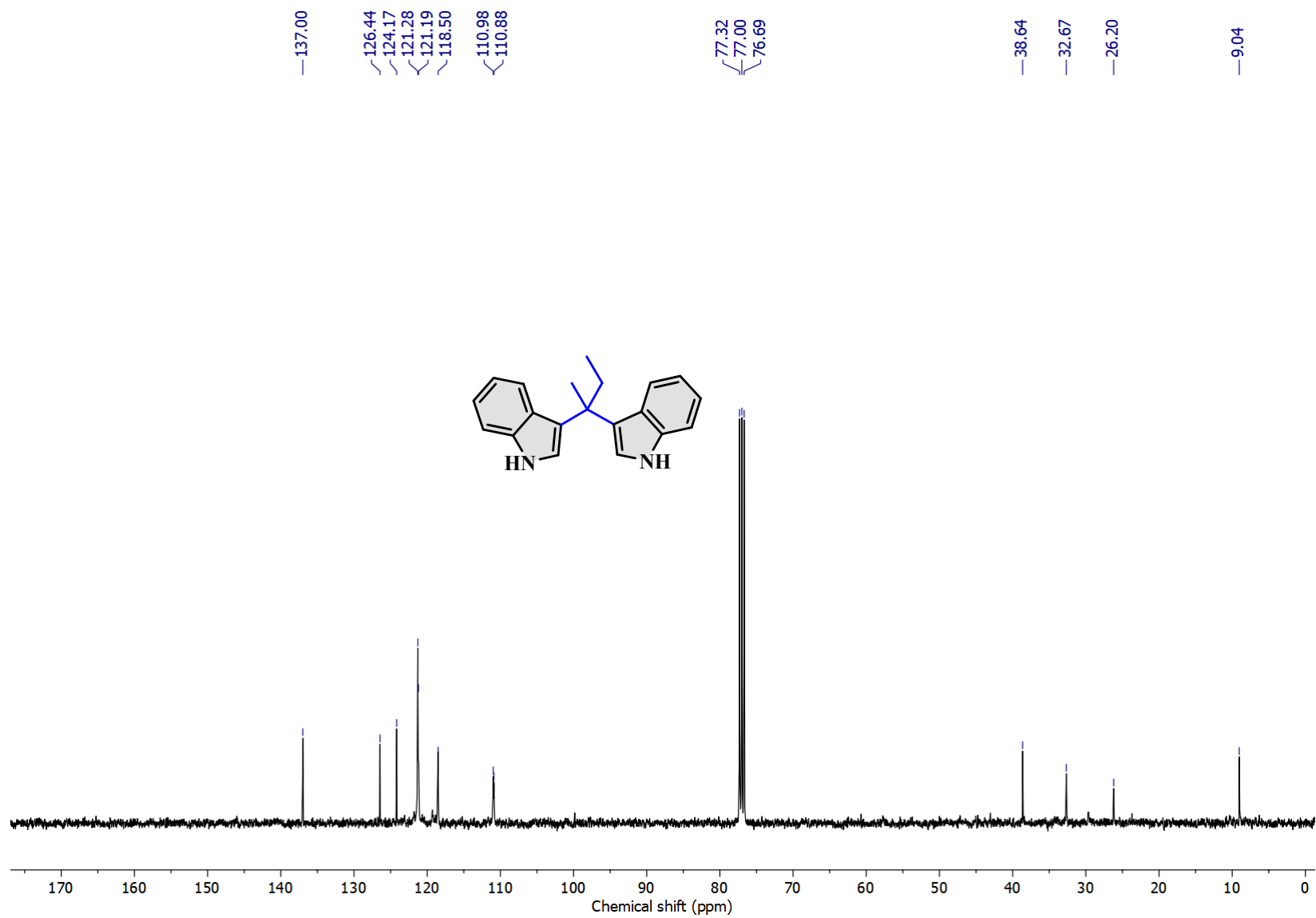


Fig. S42 100 MHz <sup>13</sup>C NMR spectrum of **(3d)<sup>4</sup>** in CDCl<sub>3</sub>

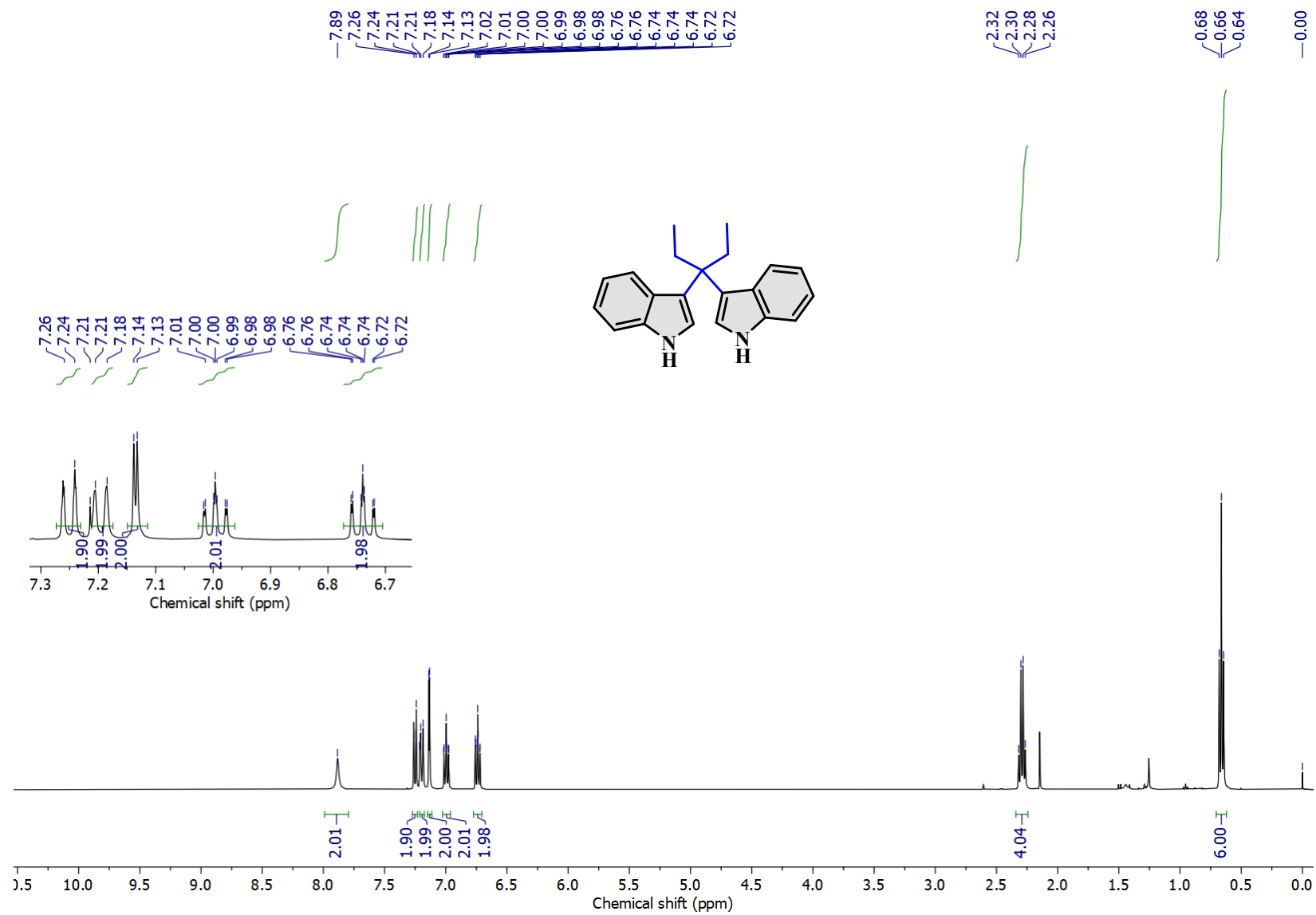


Fig. S43 400 MHz <sup>1</sup>H NMR spectrum of (3e)<sup>4</sup> in CDCl<sub>3</sub>

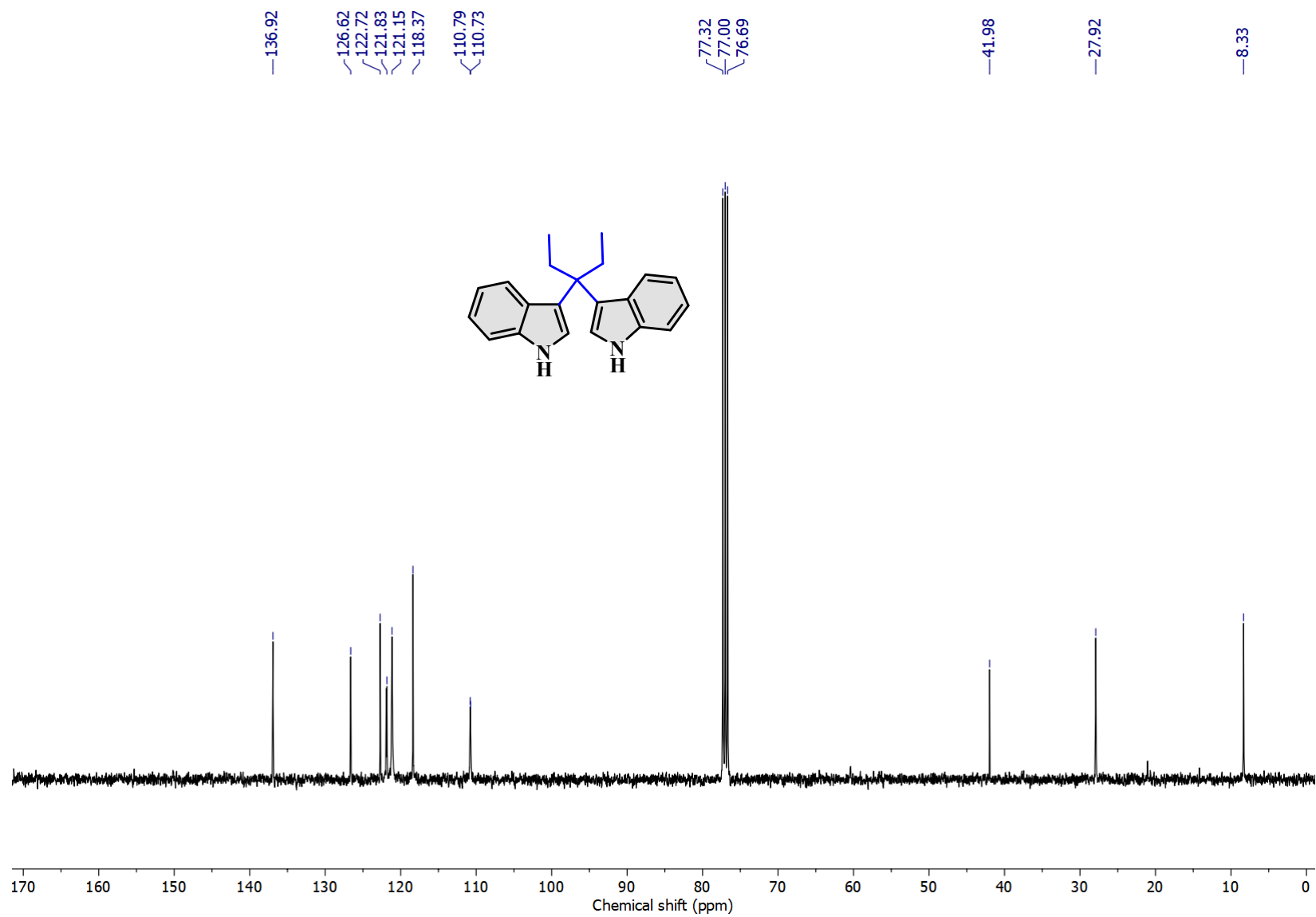


Fig. S44 100 MHz  $^{13}\text{C}$  NMR spectrum of **(3e)**<sup>4</sup> in  $\text{CDCl}_3$

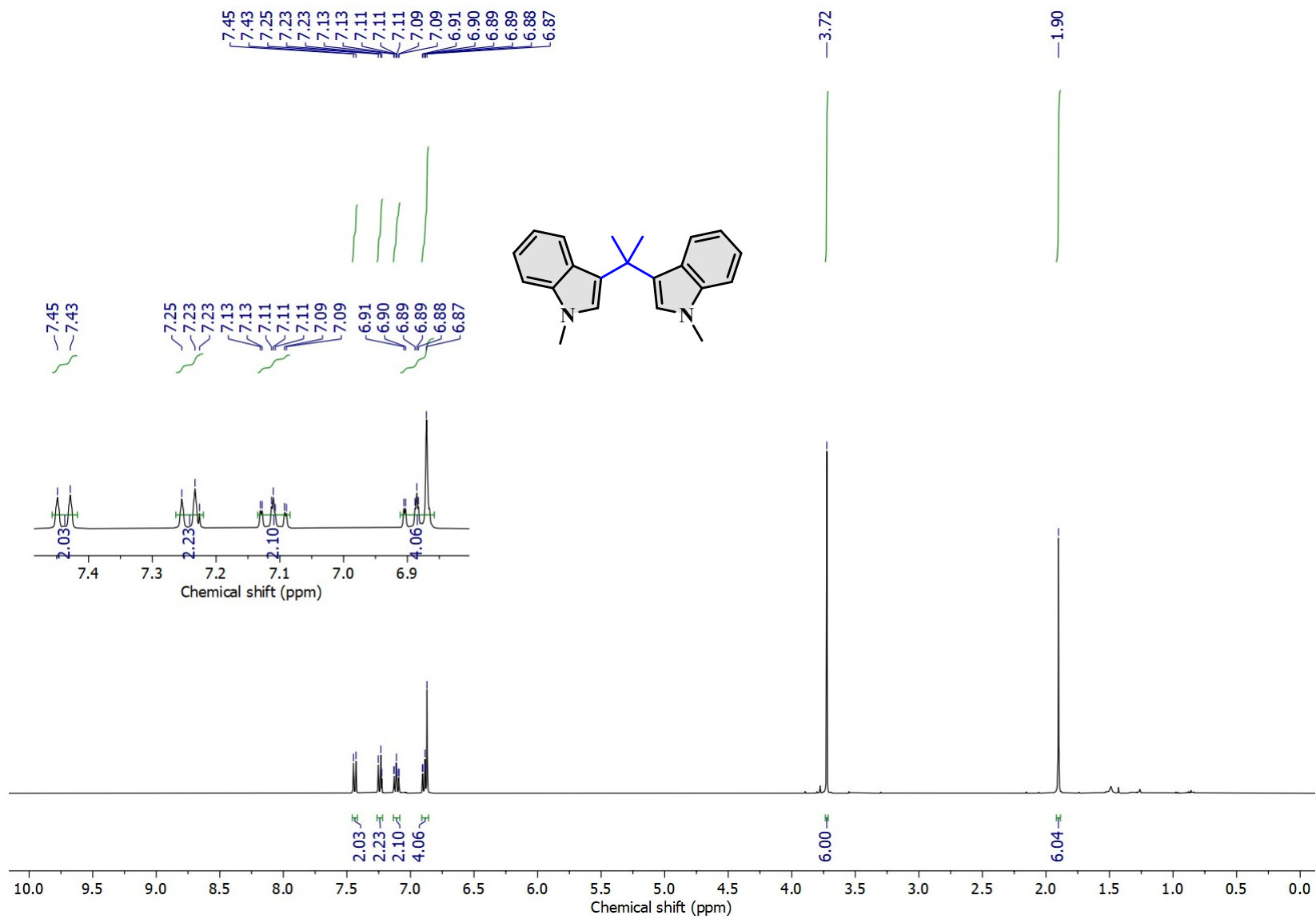
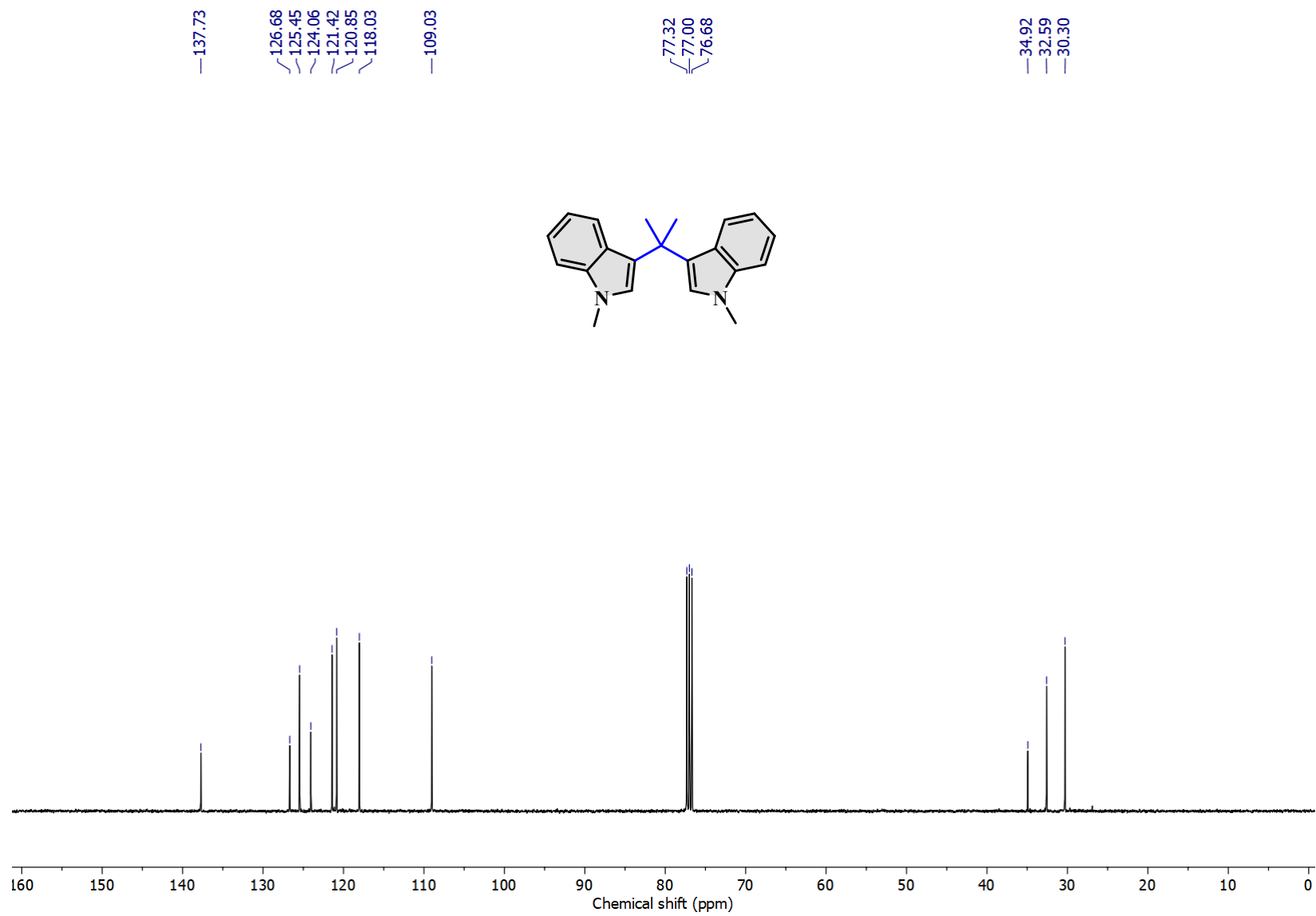


Fig. S45 400 MHz <sup>1</sup>H NMR spectrum of (3a')<sub>5</sub> in CDCl<sub>3</sub>



**Fig. S46** 100 MHz <sup>13</sup>C NMR spectrum of (3a')<sub>5</sub> in CDCl<sub>3</sub>

## 2. Copies of HRMS spectra

### Single Mass Analysis

Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

893 formula(e) evaluated with 165 results within limits (all results (up to 1000) for each mass)

Elements Used:

Mass	Calc. Mass	mDa	PPM	DBE	Formula	i-FIT	i-FIT Norm	Fit Conf %	C	H	N	O
375.2078	375.2078	0.0	0.0	5.5	C <sub>9</sub> H <sub>23</sub> N <sub>14</sub> O <sub>3</sub>	750.0	3.073	4.63	9	23	14	3
375.2073	375.2073	0.5	1.3	12.5	C <sub>24</sub> H <sub>27</sub> N <sub>2</sub> O <sub>2</sub>	755.1	8.181	0.03	24	27	2	2
375.2091	375.2091	-1.3	-3.5	-0.5	C <sub>12</sub> H <sub>31</sub> N <sub>4</sub> O <sub>9</sub>	752.7	5.768	0.31	12	31	4	9
375.2064	375.2064	1.4	3.7	0.5	C <sub>8</sub> H <sub>27</sub> N <sub>10</sub> O <sub>7</sub>	751.6	4.697	0.91	8	27	10	7
375.2104	375.2104	-2.6	-6.9	4.5	C <sub>13</sub> H <sub>27</sub> N <sub>8</sub> O <sub>5</sub>	752.1	5.199	0.55	13	27	8	5
375.2046	375.2046	3.2	8.5	13.5	C <sub>20</sub> H <sub>23</sub> N <sub>8</sub>	753.6	6.678	0.13	20	23	8	

28122020\_087\_8244

PB-456\_28122020\_004 408 (3.554)AM2 (Ar,30000,0.556,29,0.00,LS 1); Cm (376.429)

1: TOF MS ES+  
1.24e+008

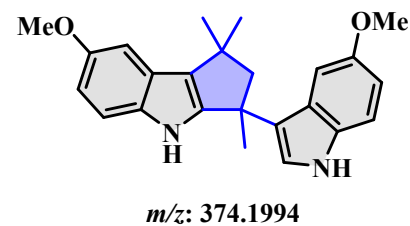
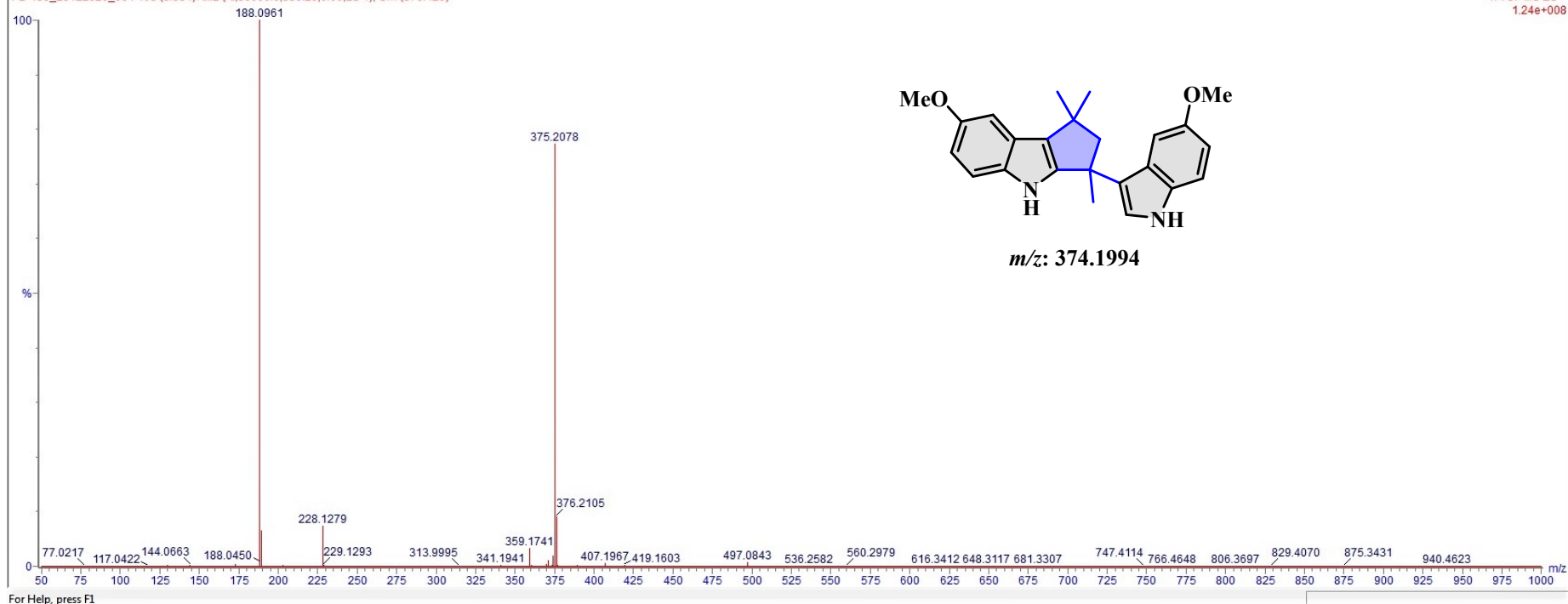


Fig. S47 HRMS spectrum of 2c



### Single Mass Analysis

Tolerance = 100.0 mDa / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

1033 formula(e) evaluated with 165 results within limits (all results (up to 1000) for each mass)

Elements Used:

Mass	Calc. Mass	mDa	PPM	DBE	Formula	i-FIT	i-FIT Norm	Fit Conf %	C	H	N	O
403.2385	403.2386	-0.1	-0.2	12.5	C26 H31 N2 O2	700.0	8.010	0.03	26	31	2	2
	403.2391	-0.6	-1.5	5.5	C11 H27 N14 O3	695.2	3.185	4.14	11	27	14	3
	403.2377	0.8	2.0	0.5	C10 H31 N10 O7	696.4	4.379	1.25	10	31	10	7
	403.2404	-1.9	-4.7	-0.5	C14 H35 N4 O9	697.8	5.866	0.28	14	35	4	9
	403.2359	2.6	6.4	13.5	C22 H27 N8	698.8	6.787	0.11	22	27	8	
	403.2417	-3.2	-7.9	4.5	C15 H31 N8 O5	697.4	5.381	0.46	15	31	8	5

28122020\_087\_8246

PB-465\_28122020\_007\_454 (3.949) AM2 (Ar,30000.0,556.32,0.00,LS 1); Cm (444:483)

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1.01e+008

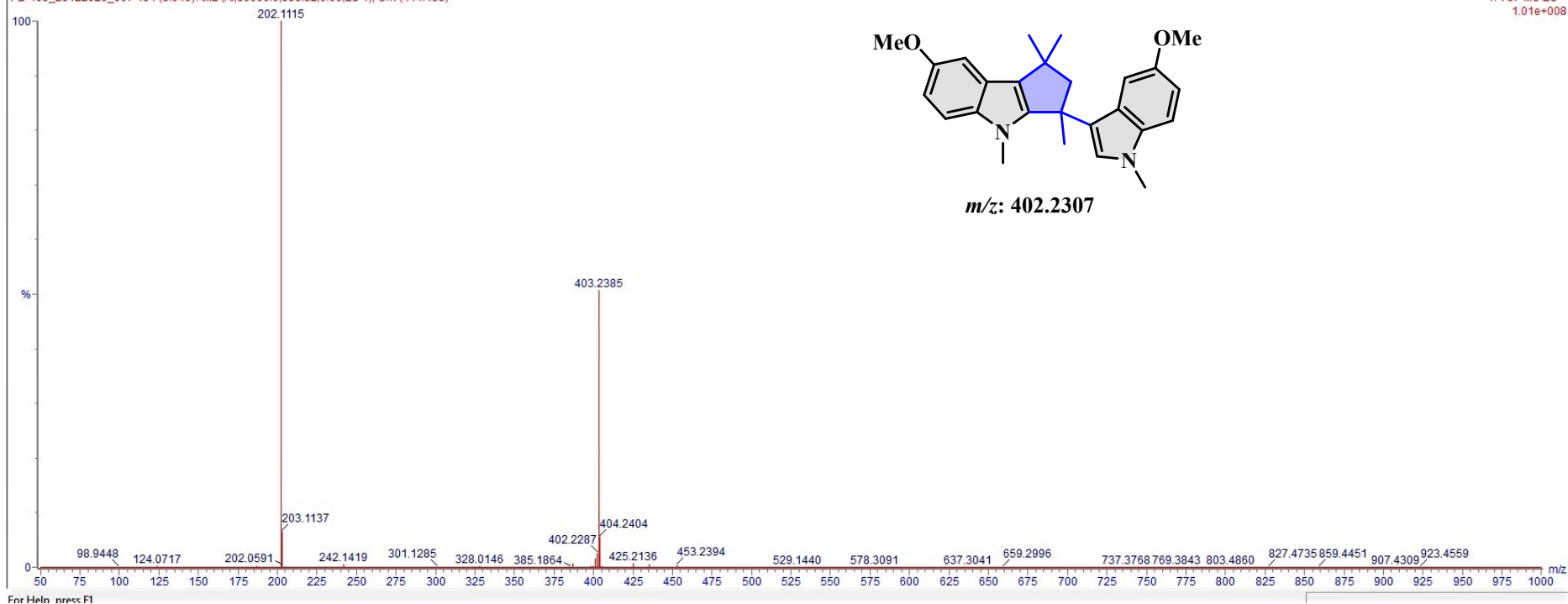
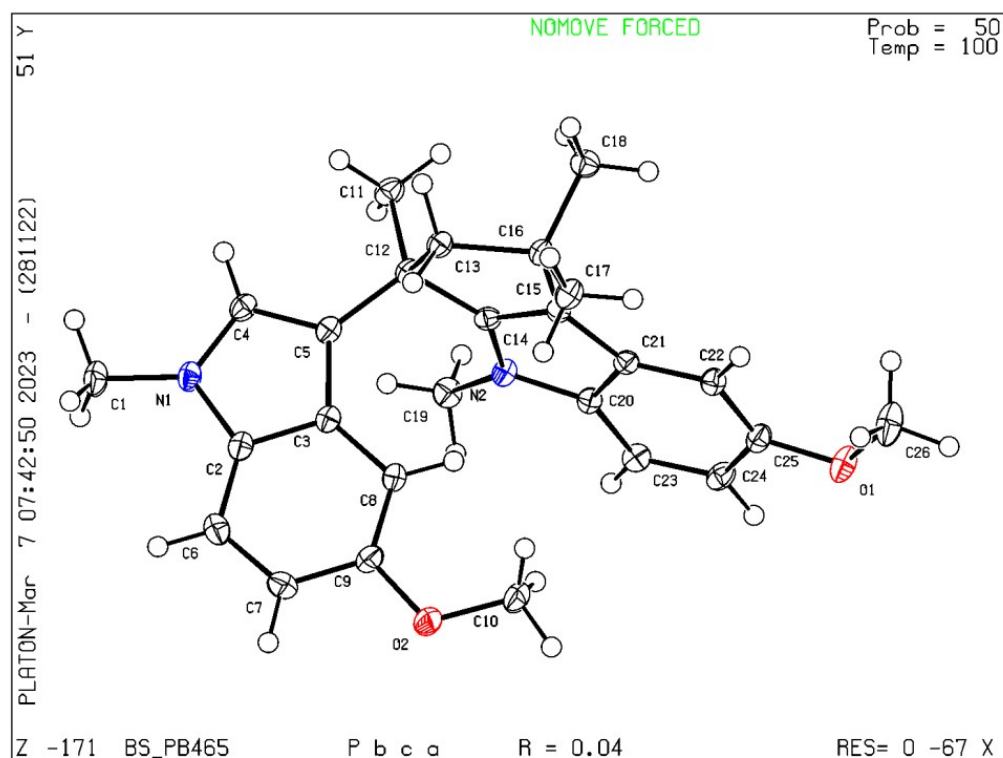


Fig. S48 HRMS spectrum of 2d

### 3. Single crystal XRD data of 2d

(CCDC 2254620)



**Fig. S49** ORTEP diagram of **2d** with 50% probability ellipsoids

**Table S1** Crystallographic parameters of structures **2d**

Crystal data	<b>2d</b>
Formula unit	C <sub>26</sub> H <sub>30</sub> N <sub>2</sub> O <sub>2</sub>
Formula weight (g mol <sup>-1</sup> )	402.52
Crystal system	orthorhombic
T [K]	100 K
<i>a</i> [Å]	8.0818(7)
<i>b</i> [Å]	16.4868(15)
<i>c</i> [Å]	32.475(3)
$\alpha$ [°]	90
$\beta$ [°]	90
$\gamma$ [°]	90
Volume [Å <sup>3</sup> ]	4327.1(7)
Space group	<i>Pbca</i>
Z	8
D <sub>cal</sub> [g/cm <sup>3</sup> ]	1.236
R <sub>1</sub> , wR <sub>2</sub>	0.0429, 0.0941
Instrument	Bruker CCD Apex II
CCDC No	CCDC 2254620

**Single crystal X-ray diffraction.** Single crystal X-ray diffractions were collected on a Bruker SMART APEX-II CCD diffractometer using Mo K $\alpha$  ( $\lambda = 0.71073 \text{ \AA}$ ) radiation. Bruker SAINT software has been employed for reducing the data and SADABS for correcting the intensities of absorption. Structure was solved and refined using SHELXL with anisotropic displacement parameters for non-H atoms. In the crystal structure H-atoms are located experimentally, whereas C–H atoms were fixed geometrically using the HFIX command in SHELX-TL. No missed symmetry observed in the final check of CIF file using PLATON. Information of crystallographic parameters for the structure is furnished in **Table S1**.

#### 4. References

1. J. Bergman, P.-O. Norrby, U. Tilstam and L. Venemalm, *Tetrahedron*, 1989, **45**, 5549-5564.
2. G. M. Shelke, V. K. Rao, R. K. Tiwari, B. S. Chhikara, K. Parang and A. Kumar, *RSC Adv.*, 2013, **3**, 22346-22352.
3. S. V. Nadkarni and J. M. Nagarkar, *Green Chem. Lett. Rev.*, 2011, **4**, 121-126.
4. C. Huo, C. Sun, C. Wang, X. Jia and W. Chang, *ACS Sustain. Chem. Eng.*, 2013, **1**, 549-553.
5. S. O. Lee, J. Choi, S. Kook and S. Y. Lee, *Org. Biomol. Chem.*, 2020, **18**, 9060-9064.