

## Facile synthesis of half-sandwich hydrido nickel complexes (NHC)Ni(H)( $\eta^5$ -C<sub>5</sub>H<sub>5</sub>)

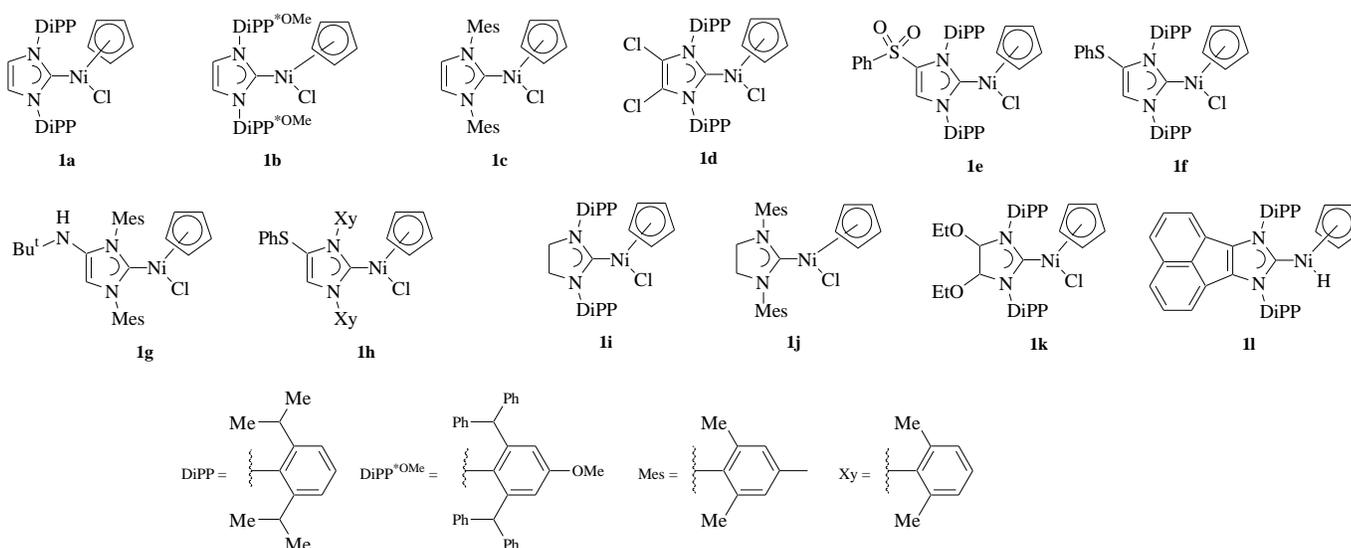
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## S1. General information and materials

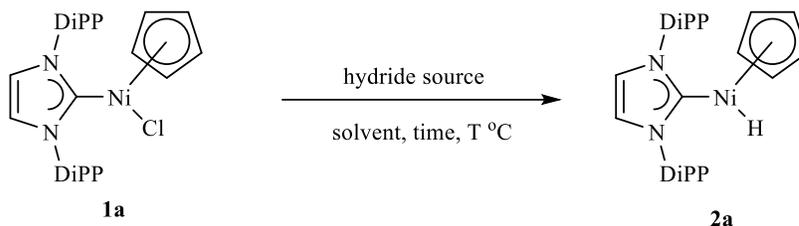
**General information.** NMR spectra were recorded on a Bruker Avance NEO 300 spectrometer at 300 MHz for <sup>1</sup>H and 75 MHz for <sup>13</sup>C. Chemical shifts ( $\delta$ , ppm) are given relative to the residual signals of benzene-*d*<sub>6</sub> protons (7.16 ppm for <sup>1</sup>H NMR) or carbon signals in benzene-*d*<sub>6</sub> (128.06 ppm for <sup>13</sup>C NMR). Solvents were purified and dried according to standard methods and stored over activated 3Å molecular sieves prior to use. Glassware was dried at 120 °C in an oven for at least 3 h before the use. Elemental analyses were performed using a Perkin Elmer 2400 elemental analyzer. Complexes **1a**,<sup>S1</sup> **1b**,<sup>S2</sup> **1c**,<sup>S1</sup> **1d**,<sup>S3</sup> **1e**,<sup>S4</sup> **1f**,<sup>S5</sup> **1g**,<sup>S6</sup> **1h**,<sup>S5</sup> **1i,j**,<sup>S7</sup> **1k**<sup>S4</sup> and **1l**<sup>S8</sup> were synthesized as described in the literature. All other chemicals were purchased from commercial sources.



**Figure S1.** Overview of the [(NHC)Ni(Cl)( $\eta^5$ -Cp)] complexes used in this study.

## S2. Extended experimental data

**Table S1.** Effect of reaction conditions on the yield of **2a** complex.<sup>a</sup>



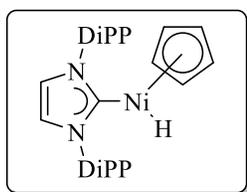
Entry	Hydride source (eq.)	Solvent	<i>t</i> /h	<i>T</i> /°C	Yield of <b>2a</b> , % <sup>b</sup>
1	Et <sub>3</sub> SiH (10)	Dioxane	16	25	0
2	(EtO) <sub>3</sub> SiH (10)	Dioxane	16	25	0
3	Et <sub>3</sub> SiH (10) <sup>c</sup>	Dioxane	16	25	19
4	(EtO) <sub>3</sub> SiH (10) <sup>c</sup>	Dioxane	16	25	21
5	NaH (10)	Dioxane	16	25	7
6	CaH <sub>2</sub> (10)	Dioxane	16	25	0
7	CaH <sub>2</sub> (10)	Dioxane	16	110	0
8	NaBH <sub>4</sub> (10)	Dioxane	16	25	45
9	LiAlH <sub>4</sub> (10)	Dioxane	16	25	51
10	NaAlH <sub>4</sub> (10)	Dioxane	16	25	58
11	NaAlH <sub>4</sub> (10)	Dioxane	16	40	64
12	NaAlH <sub>4</sub> (10)	Dioxane	16	60	56
13	NaAlH <sub>4</sub> (10)	Dioxane	16	80	31
14	NaAlH <sub>4</sub> (10)	Dioxane	16	15	37
15	NaAlH <sub>4</sub> (10)	Dioxane	2	40	94
16	NaAlH <sub>4</sub> (10)	Dioxane	1	40	45
17	NaAlH <sub>4</sub> (5)	Dioxane	2	40	58
18	NaAlH <sub>4</sub> (20)	Dioxane	2	40	87
19	NaAlH <sub>4</sub> (10)	THF	2	40	81
20	NaAlH <sub>4</sub> (10)	Toluene	2	40	76
21	NaAlH <sub>4</sub> (10)	Benzene	2	40	68

<sup>a</sup> Reaction conditions: **1a** (0.05 mmol), hydride source (5–20 eq.), solvent (1 mL), 15–110 °C, 1–16 h. <sup>b</sup> The yields were determined by <sup>1</sup>H NMR spectroscopy following centrifugation of the reaction mixtures. <sup>c</sup> In the presence of the Bu<sup>t</sup>ONa (1 eq).

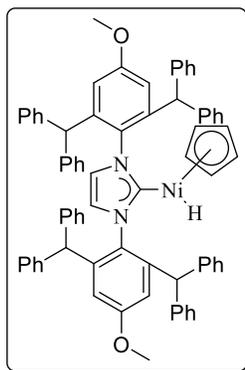
### S3. Characterization of synthesized compounds

#### Synthesis of complexes (NHC)Ni(Cp)H.

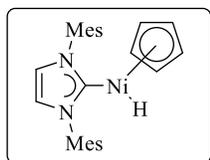
An oven-dried vial equipped with a magnetic stir bar and a septum was charged in air with a complex **1a-1** (0.25 mmol), NaAlH<sub>4</sub> (135 mg, 2.5 mmol) and 1,4-dioxane (2 mL). The mixture was degassed and purged with argon (three cycles) using standard Schlenk techniques and was stirred vigorously at 40 °C for 2 h. During the reaction, slight lightening of the mixture was observed without significant gas evolution. After cooling to room temperature, the reaction mixture was filtered in argon atmosphere through a short Celite 545 pad (10×10 mm). The celite was additionally washed with 1,4-dioxane (2 ml). The filtrate combined with the washings was then rotary evaporated under reduced pressure and an argon flow to yield the desired pure red crystalline product which was collected and used further without additional purification. *It is important to note that the resulting hydride complexes should be stored in a refrigerator under argon atmosphere to prevent decomposition.*



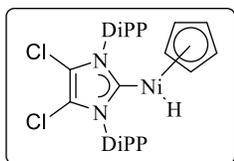
**Complex 2a.** Yield 117 mg (91 %), red powder. <sup>1</sup>H NMR (C<sub>6</sub>D<sub>6</sub>, 300 MHz): δ -23.62 (s, 1H), 1.08 (d, *J* = 6.9 Hz, 12H), 1.45 (d, *J* = 6.9 Hz, 12H), 2.92 (sept, *J* = 6.9 Hz, 4H), 4.92 (s, 5H), 6.45 (s, 2H), 7.12-7.15 (m, 4H), 7.22-7.29 (m, 2H). <sup>13</sup>C NMR (C<sub>6</sub>D<sub>6</sub>, 75 MHz): δ 23.2, 25.0, 28.9, 87.0, 122.5, 124.0, 129.7, 138.7, 146.3, 188.6. The spectral characteristics of the product obtained are similar to those described in the literature.<sup>S9</sup>



**Complex 2b.** Yield 235 mg (88 %), brown powder. <sup>1</sup>H NMR (C<sub>6</sub>D<sub>6</sub>, 300 MHz): δ -22.5 (s, 1H), 3.25 (s, 6H), 5.34 (s, 5H), 5.44 (s, 2H), 5.96 (s, 4H), 7.06-7.13 (m, 18H), 7.24-7.34 (m, 10H), 7.36-7.45 (m, 8H), 7.88-7.98 (m, 8H). <sup>13</sup>C NMR (C<sub>6</sub>D<sub>6</sub>, 75 MHz): δ 52.4, 54.7, 88.2, 114.9, 122.6, 126.5, 127.1, 128.6, 129.7, 129.9, 130.9, 133.8, 143.9, 144.1, 144.3, 160.2, 188.8. Anal. calcd. for C<sub>74</sub>H<sub>62</sub>N<sub>2</sub>NiO<sub>2</sub> (%): C, 83.07; H, 5.84; N, 2.62. Found (%): C, 83.16; H, 5.89; N, 2.65.

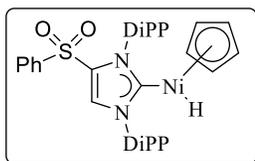


**Complex 2c.** Yield 81 mg (76 %), red powder. <sup>1</sup>H NMR (C<sub>6</sub>D<sub>6</sub>, 300 MHz): δ -23.5 (s, 1H), 2.12 (s, 18H), 4.99 (s, 5H), 6.14 (s, 2H), 6.80 (br s, 4H). <sup>13</sup>C NMR (C<sub>6</sub>D<sub>6</sub>, 75 MHz): δ 18.3, 21.1, 86.8, 121.0, 129.2, 135.8, 138.3, 138.6, 185.4. The spectral characteristics of the product obtained are similar to those described in the literature.<sup>S10</sup>

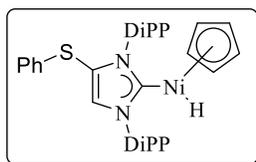


**Complex 2d.** Yield 115 mg (79 %), red powder. <sup>1</sup>H NMR (C<sub>6</sub>D<sub>6</sub>, 300 MHz): δ -23.36 (s, 1H), 1.17 (d, *J* = 6.9 Hz, 12H), 1.49 (d, *J* = 6.9 Hz, 12H), 2.89 (sept, *J* = 6.9 Hz, 4H),

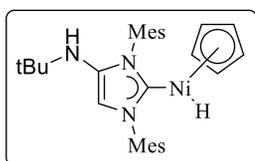
4.85 (s, 5H), 7.12-7.15 (m, 2H), 7.17-7.20 (m, 2H), 7.24-7.30 (m, 2H).  $^{13}\text{C}$  NMR ( $\text{C}_6\text{D}_6$ , 75 MHz):  $\delta$  23.7, 24.1, 29.3, 87.5, 116.6, 124.4, 130.6, 135.8, 146.8, 190.6. Anal. calcd. for  $\text{C}_{32}\text{H}_{40}\text{Cl}_2\text{N}_2\text{Ni}$  (%): C, 66.01; H, 6.92; N, 4.81. Found (%): C, 66.09; H, 6.88; N, 4.85.



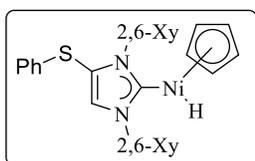
**Complex 2e.** Yield 117 mg (72 %), brown powder.  $^1\text{H}$  NMR ( $\text{C}_6\text{D}_6$ , 300 MHz):  $\delta$  -23.05 (s, 1H), 0.96 (d,  $J = 6.8$  Hz, 6H), 0.98 (d,  $J = 6.8$  Hz, 6H), 1.34 (d,  $J = 6.8$  Hz, 6H), 1.60 (d,  $J = 6.8$  Hz, 6H), 2.72 (sept,  $J = 6.8$  Hz, 2H), 2.92 (sept,  $J = 6.8$  Hz, 2H), 4.78 (s, 5H), 6.65-6.84 (m, 3H), 7.03-7.12 (m, 4H), 7.20-7.30 (m, 2H), 7.35-7.40 (m, 2H), 7.49 (s, 1H).  $^{13}\text{C}$  NMR ( $\text{C}_6\text{D}_6$ , 75 MHz):  $\delta$  22.7, 22.8, 24.8, 25.5, 29.0, 29.8, 87.8, 124.3, 124.3, 128.6, 129.1, 130.3, 130.4, 131.1, 133.5, 134.0, 136.3, 137.9, 140.8, 146.1, 146.9, 197.3. Anal. calcd. for  $\text{C}_{38}\text{H}_{46}\text{N}_2\text{NiO}_2\text{S}$  (%): C, 69.84; H, 7.09; N, 4.29. Found (%): C, 69.92; H, 7.05; N, 4.26.



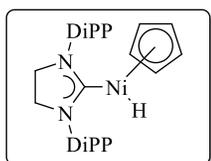
**Complex 2f.** Yield 126 mg (81 %), brown powder.  $^1\text{H}$  NMR (300 MHz,  $\text{C}_6\text{D}_6$ ):  $\delta$  -23.57 (s, 1H), 1.04 (d,  $J = 7.0$  Hz, 6H), 1.08 (d,  $J = 7.0$  Hz, 6H), 1.46 (d,  $J = 7.0$  Hz, 6H), 1.55 (d,  $J = 7.0$  Hz, 6H), 2.74 (sept,  $J = 7.0$  Hz, 2H), 3.01 (sept,  $J = 7.0$  Hz, 2H), 4.88 (s, 5H), 6.68 (s, 1H), 6.81-6.92 (m, 3H), 7.03-7.08 (m, 2H), 7.10-7.13 (m, 2H), 7.13-7.15 (m, 2H), 7.21-7.29 (m, 2H).  $^{13}\text{C}$  NMR (75 MHz,  $\text{C}_6\text{D}_6$ ):  $\delta$  23.1, 24.0, 24.4, 25.1, 29.0, 29.3, 87.4, 124.1, 124.2, 124.2, 127.0, 128.8, 129.4, 129.9, 130.0, 135.1, 136.7, 138.5, 146.2, 147.0, 192.1. Anal. calcd. for  $\text{C}_{38}\text{H}_{46}\text{N}_2\text{NiS}$  (%): C, 73.43; H, 7.46; N, 4.51. Found (%): C, 73.51; H, 7.42; N, 4.56.



**Complex 2g.** Yield 93 mg (74 %), brown powder.  $^1\text{H}$  NMR ( $\text{C}_6\text{D}_6$ , 300 MHz):  $\delta$  -23.53 (s, 1H), 0.81 (s, 9H), 2.08 (s, 3H), 2.16 (s, 3H), 2.24 (s, 6H), 2.29 (s, 6H), 5.03 (s, 5H), 5.82 (s, 1H), 6.82 (s, 2H), 6.87 (s, 2H).  $^{13}\text{C}$  NMR ( $\text{C}_6\text{D}_6$ , 75 MHz):  $\delta$  18.4, 18.5, 21.2, 21.2, 28.8, 51.1, 86.7, 101.0, 129.1, 129.6, 134.2, 136.1, 137.3, 138.0, 139.0, 139.2, 177.8. Anal. calcd. for  $\text{C}_{30}\text{H}_{39}\text{N}_3\text{Ni}$  (%): C, 72.01; H, 7.86; N, 8.40. Found (%): C, 72.13; H, 7.80; N, 8.44.

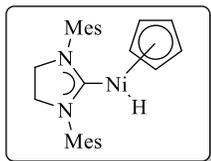


**Complex 2h.** Yield 87 mg (68 %), brown powder.  $^1\text{H}$  NMR ( $\text{C}_6\text{D}_6$ , 300 MHz):  $\delta$  -23.02 (s, 1H), 1.89 (s, 6H), 2.12 (s, 6H), 4.89 (s, 5H), 6.14 (s, 1H), 6.80-6.86 (m, 3H), 6.87-6.99 (m, 6H), 7.03-7.10 (m, 2H).  $^{13}\text{C}$  NMR ( $\text{C}_6\text{D}_6$ , 75 MHz):  $\delta$  18.4, 27.2, 86.9, 123.6, 127.4, 129.0, 129.1, 131.5, 133.8, 136.0, 137.3, 138.8, 140.7, 189.7. Anal. calcd. for  $\text{C}_{30}\text{H}_{30}\text{N}_2\text{NiS}$  (%): C, 70.74; H, 5.94; N, 5.50. Found (%): C, 70.81; H, 5.98; N, 5.54.

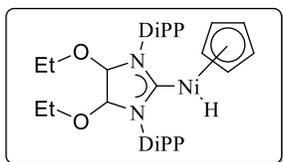


**Complex 2i.** Yield 115 mg (89 %), brown powder.  $^1\text{H}$  NMR ( $\text{C}_6\text{D}_6$ , 300 MHz):  $\delta$  -23.03 (s, 1H), 1.17 (d,  $J = 7.1$  Hz, 12H), 1.52 (d,  $J = 7.1$  Hz, 12H), 3.22 (sept,  $J = 7.1$  Hz, 4H), 3.40 (s, 4H), 4.85 (s, 5H), 7.10-7.15 (m, 4H), 7.18-7.26 (m, 2H).  $^{13}\text{C}$  NMR ( $\text{C}_6\text{D}_6$ , 75 MHz):  $\delta$  24.0, 25.6, 28.9, 52.9, 87.4, 124.4, 139.3, 147.4, 214.9. Anal. calcd. for  $\text{C}_{32}\text{H}_{44}\text{N}_2\text{Ni}$  (%): C, 74.57; H, 8.61; N, 5.44. Found (%): C, 74.67; H, 8.65; N, 5.49.

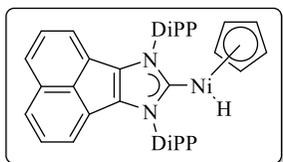
$\text{C}_{32}\text{H}_{44}\text{N}_2\text{Ni}$  (%): C, 74.57; H, 8.61; N, 5.44. Found (%): C, 74.67; H, 8.65; N, 5.49.



**Complex 2j.** Yield 79 mg (73 %), brown powder.  $^1\text{H}$  NMR ( $\text{C}_6\text{D}_6$ , 300 MHz):  $\delta$  -22.56, 2.13 (s, 6H), 2.28 (s, 12H), 3.10 (s, 4H), 4.92 (s, 5H), 6.83 (s, 4H).  $^{13}\text{C}$  NMR ( $\text{C}_6\text{D}_6$ , 75 MHz):  $\delta$  18.3, 21.1, 49.8, 87.1, 129.5, 136.7, 137.4, 139.0, 212.9. Anal. calcd. for  $\text{C}_{26}\text{H}_{32}\text{N}_2\text{Ni}$  (%): C, 72.41; H, 7.48; N, 6.50. Found (%): C, 72.49; H, 7.53; N, 6.54.

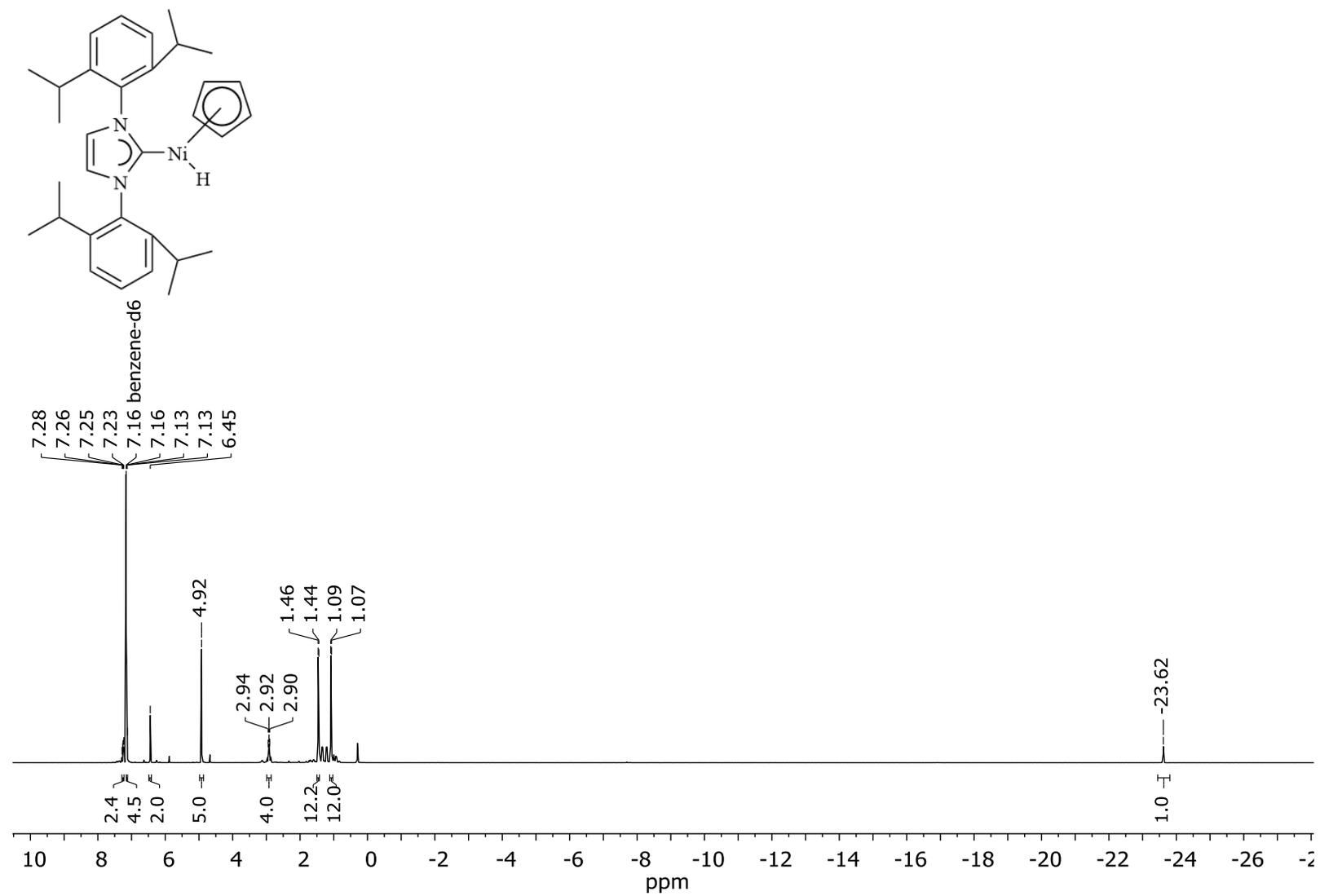


**Complex 2k.** Yield 121 mg (84 %), brown powder.  $^1\text{H}$  NMR ( $\text{C}_6\text{D}_6$ , 300 MHz):  $\delta$  -22.26 (s, 1H), 0.87 (t,  $J = 7.1$  Hz, 6H), 1.20 (d,  $J = 7.0$  Hz, 6H), 1.38 (d,  $J = 7.1$  Hz, 6H), 1.47 (d,  $J = 7.0$  Hz, 6H), 1.72 (d,  $J = 7.1$  Hz, 6H), 2.89-2.98 (m, 2H), 3.01-3.09 (m, 2H), 3.12-3.20 (m, 2H), 3.76 (sept,  $J = 7.0$  Hz, 2H), 4.79 (s, 2H), 4.91 (s, 5H), 4.82 (s, 2H), 7.06-7.12 (m, 2H), 7.23-7.24 (m, 4H).  $^{13}\text{C}$  NMR ( $\text{C}_6\text{D}_6$ , 75 MHz):  $\delta$  14.3, 15.1, 24.2, 25.2, 25.6, 26.0, 28.2, 28.9, 64.7, 88.3, 98.6, 124.3, 124.8, 129.3, 137.8, 147.0, 149.9, 204.2. Anal. calcd. for  $\text{C}_{34}\text{H}_{48}\text{N}_2\text{NiO}_2$  (%): C, 70.96; H, 8.41; N, 4.87. Found (%): C, 71.08; H, 8.38; N, 4.83.

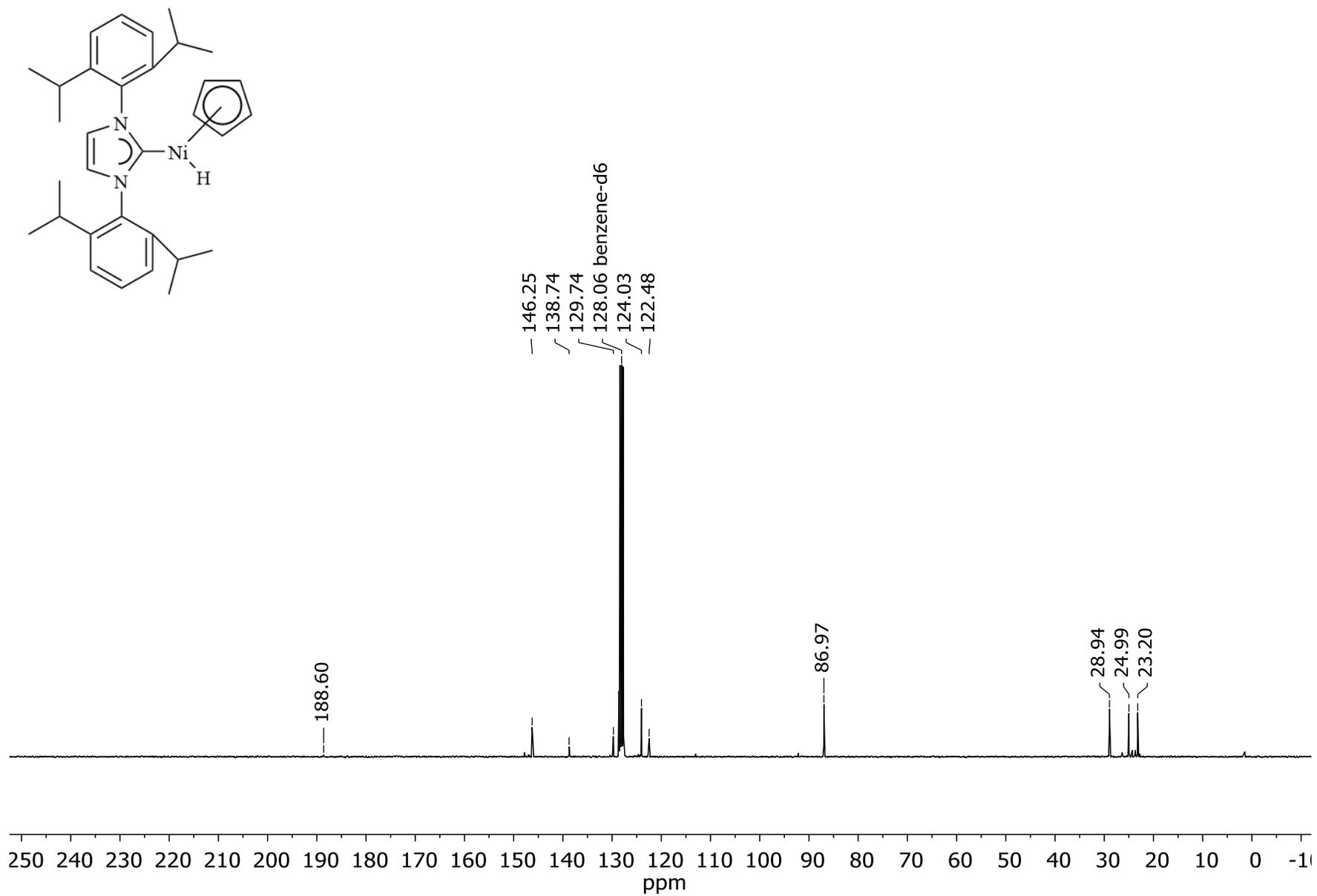


**Complex 2l.** Yield 124 mg (78 %), brown powder.  $^1\text{H}$  NMR ( $\text{C}_6\text{D}_6$ , 300 MHz):  $\delta$  -22.87 (s, 1H) 1.02 (d,  $J = 6.7$  Hz, 12H), 1.50 (d,  $J = 6.7$  Hz, 12H), 3.28 (sept,  $J = 6.7$  Hz, 4H), 5.00 (s, 5H), 6.72-6.78 (m, 2H), 6.82-6.90 (m, 2H), 7.17-7.22 (m, 2H), 7.24-7.32 (m, 4H), 7.33-7.41 (m, 2H).  $^{13}\text{C}$  NMR ( $\text{C}_6\text{D}_6$ , 75 MHz): 23.5, 24.5, 29.3, 87.3, 120.4, 124.4, 127.3, 127.5, 127.6, 130.1, 130.6, 137.4, 138.7, 146.5, 197.0. Anal. calcd. for  $\text{C}_{42}\text{H}_{46}\text{N}_2\text{Ni}$  (%): C, 79.13; H, 7.27; N, 4.39. Found (%): C, 79.18; H, 7.34; N, 4.42.

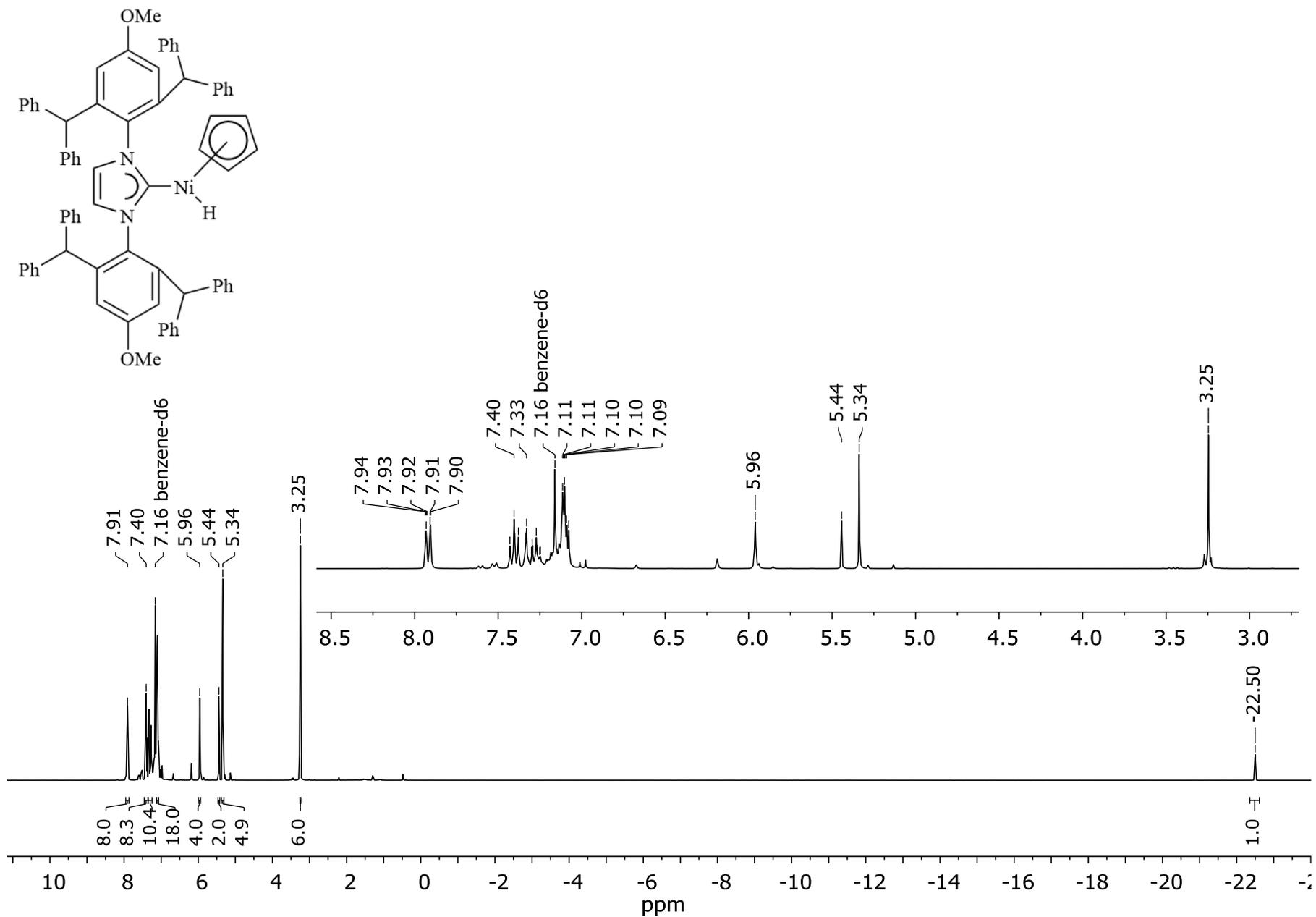
### S3. $^1\text{H}$ and $^{13}\text{C}$ NMR spectra



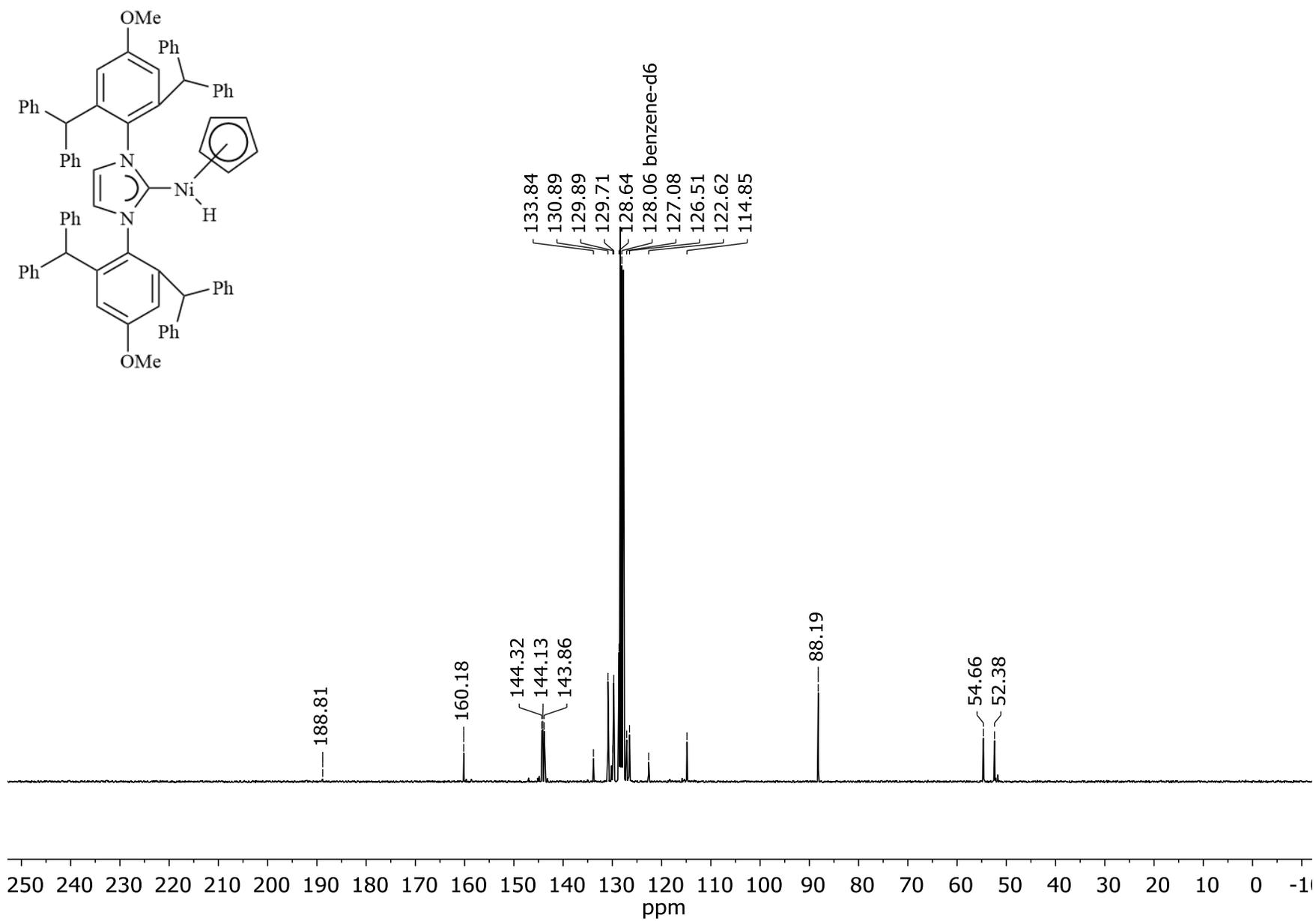
**Figure S2.**  $^1\text{H}$  NMR spectrum of compound **2a** ( $\text{C}_6\text{D}_6$ , 300 MHz)



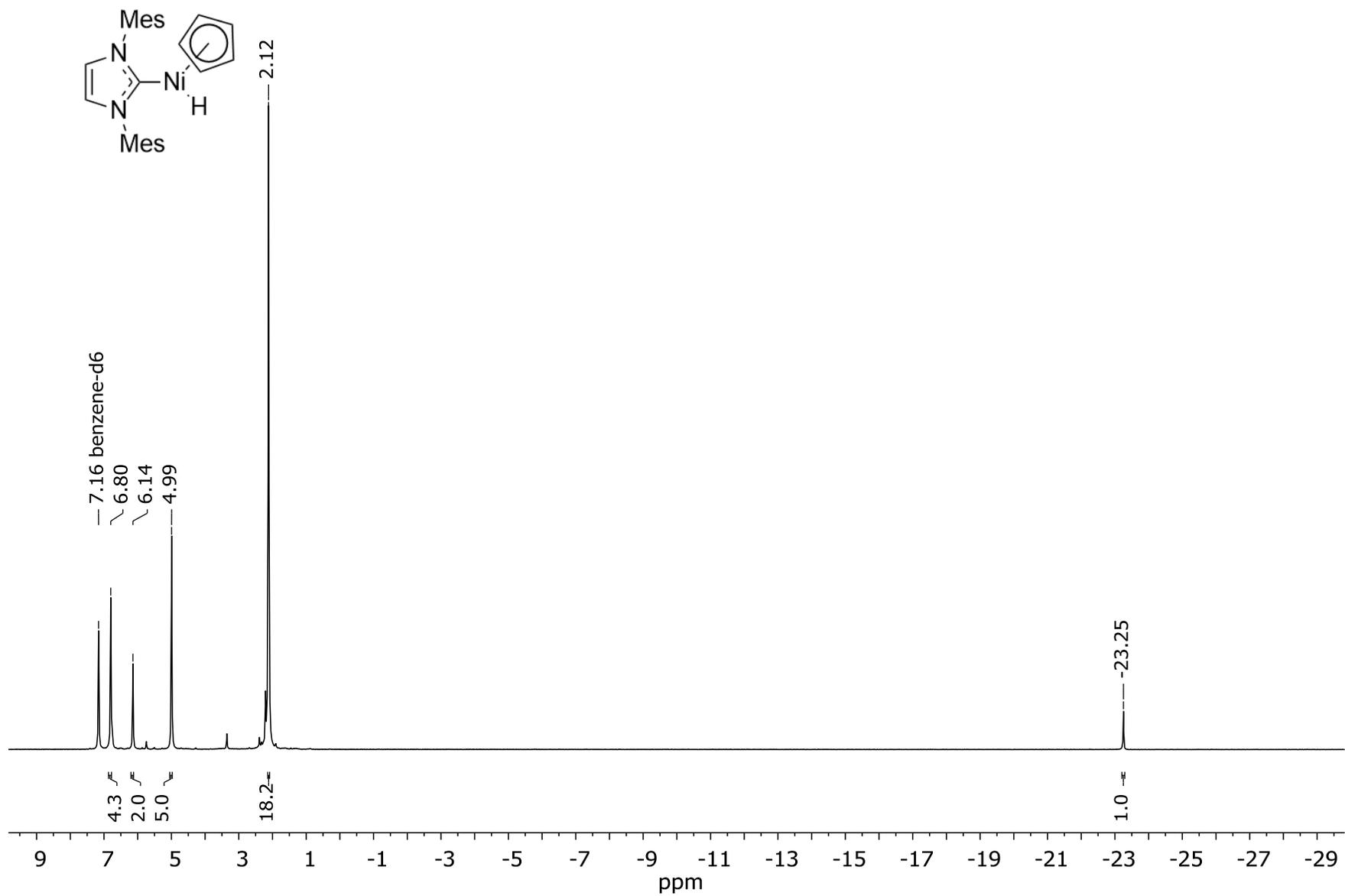
**Figure S3.**  $^{13}\text{C}$  NMR spectrum of compound **2a** ( $\text{C}_6\text{D}_6$ , 75 MHz)



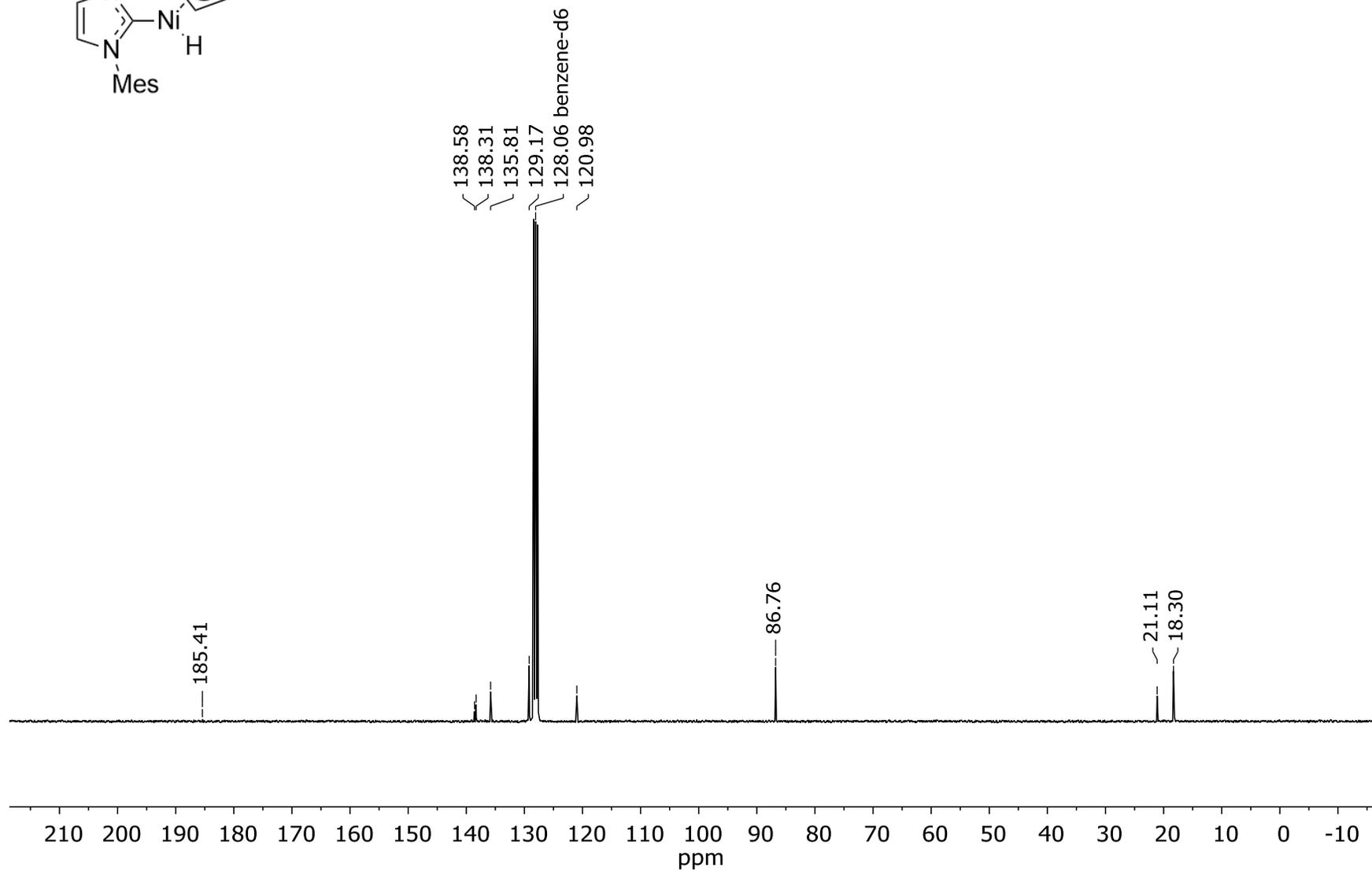
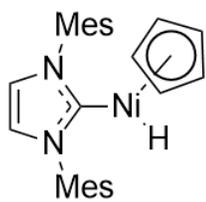
**Figure S4.**  $^1\text{H}$  NMR spectrum of compound **2b** ( $\text{C}_6\text{D}_6$ , 300 MHz)



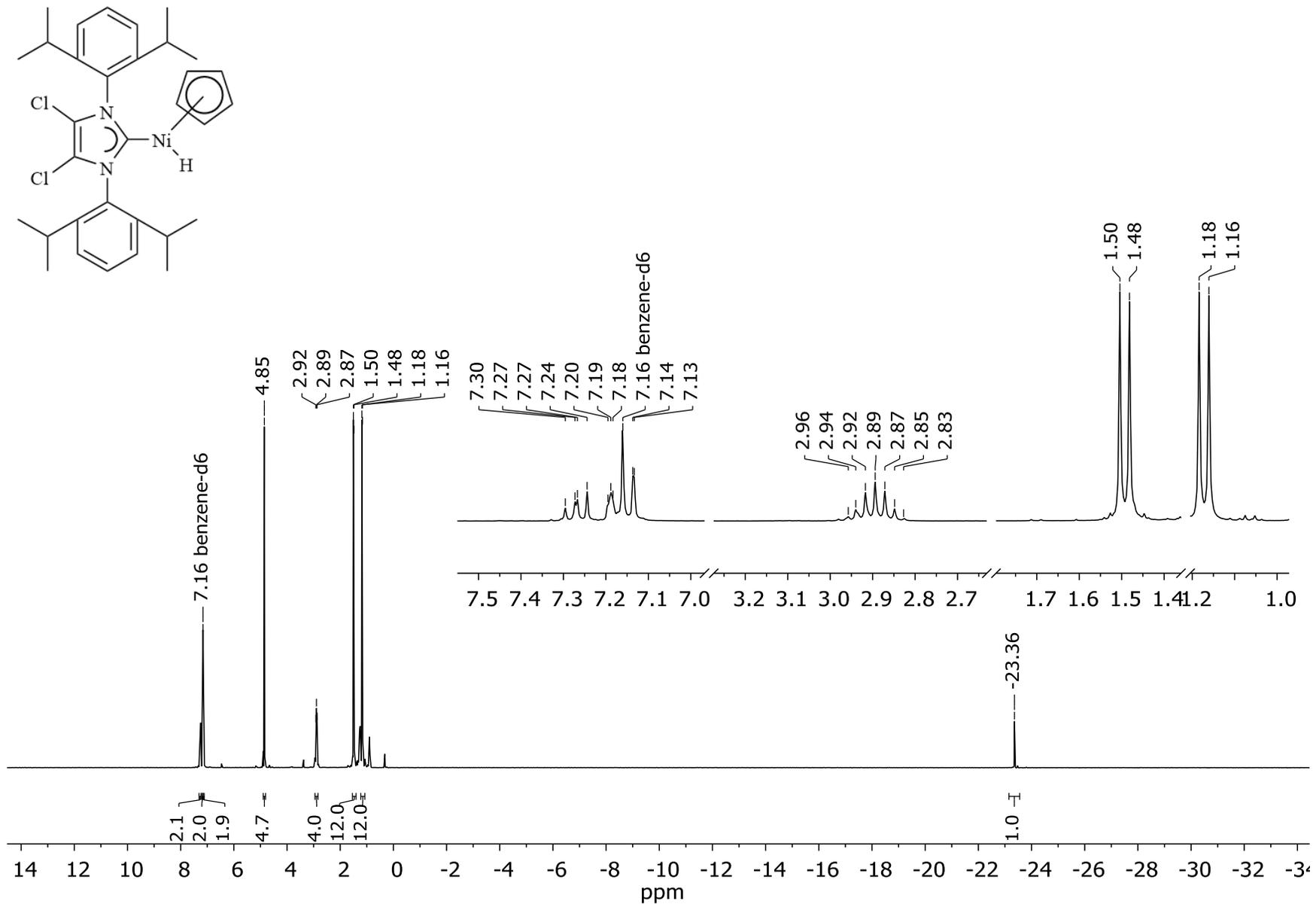
**Figure S5.**  $^{13}\text{C}$  NMR spectrum of compound **2b** ( $\text{C}_6\text{D}_6$ , 75 MHz)



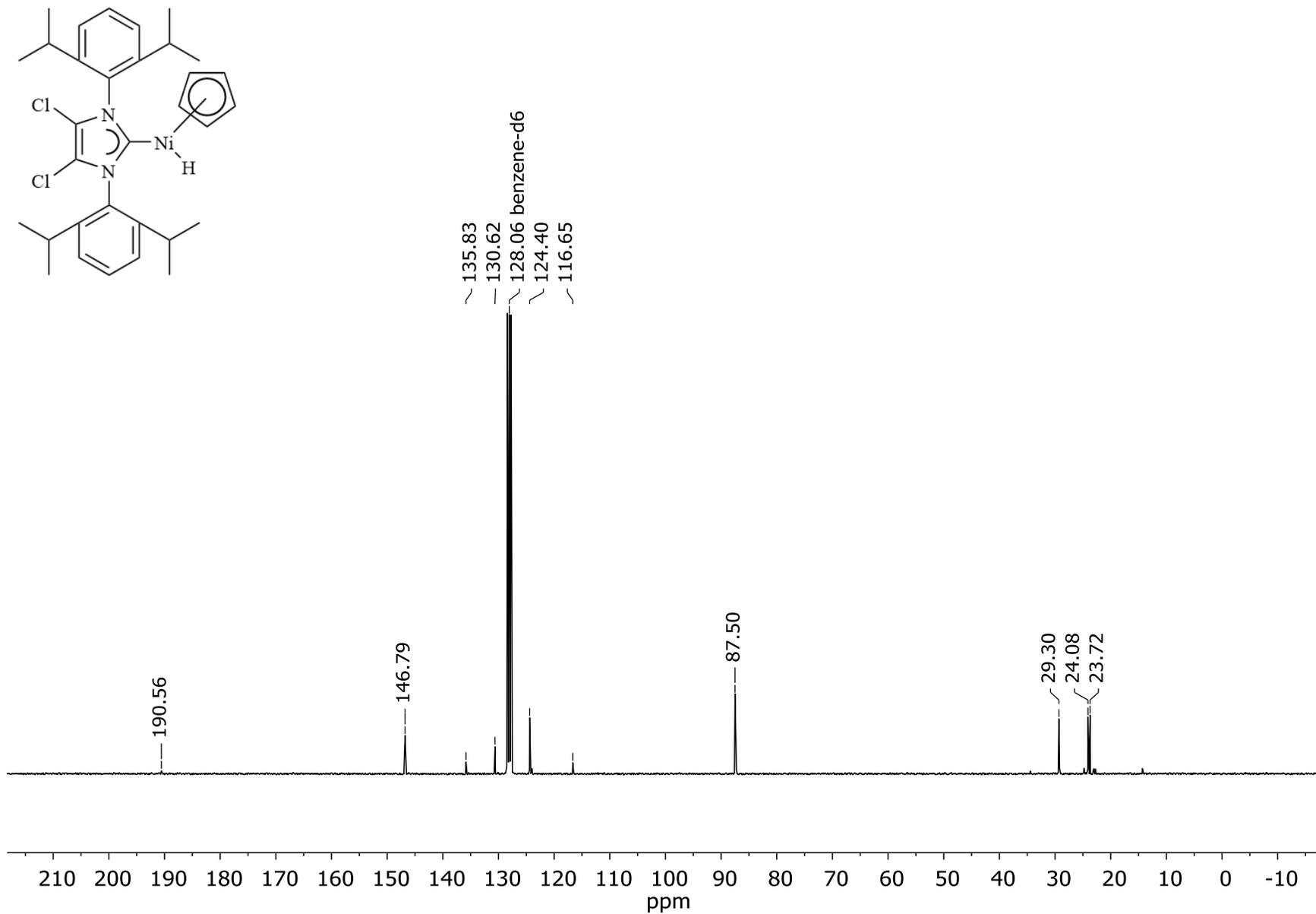
**Figure S6.**  $^1\text{H}$  NMR spectrum of compound **2c** ( $\text{C}_6\text{D}_6$ , 300 MHz)



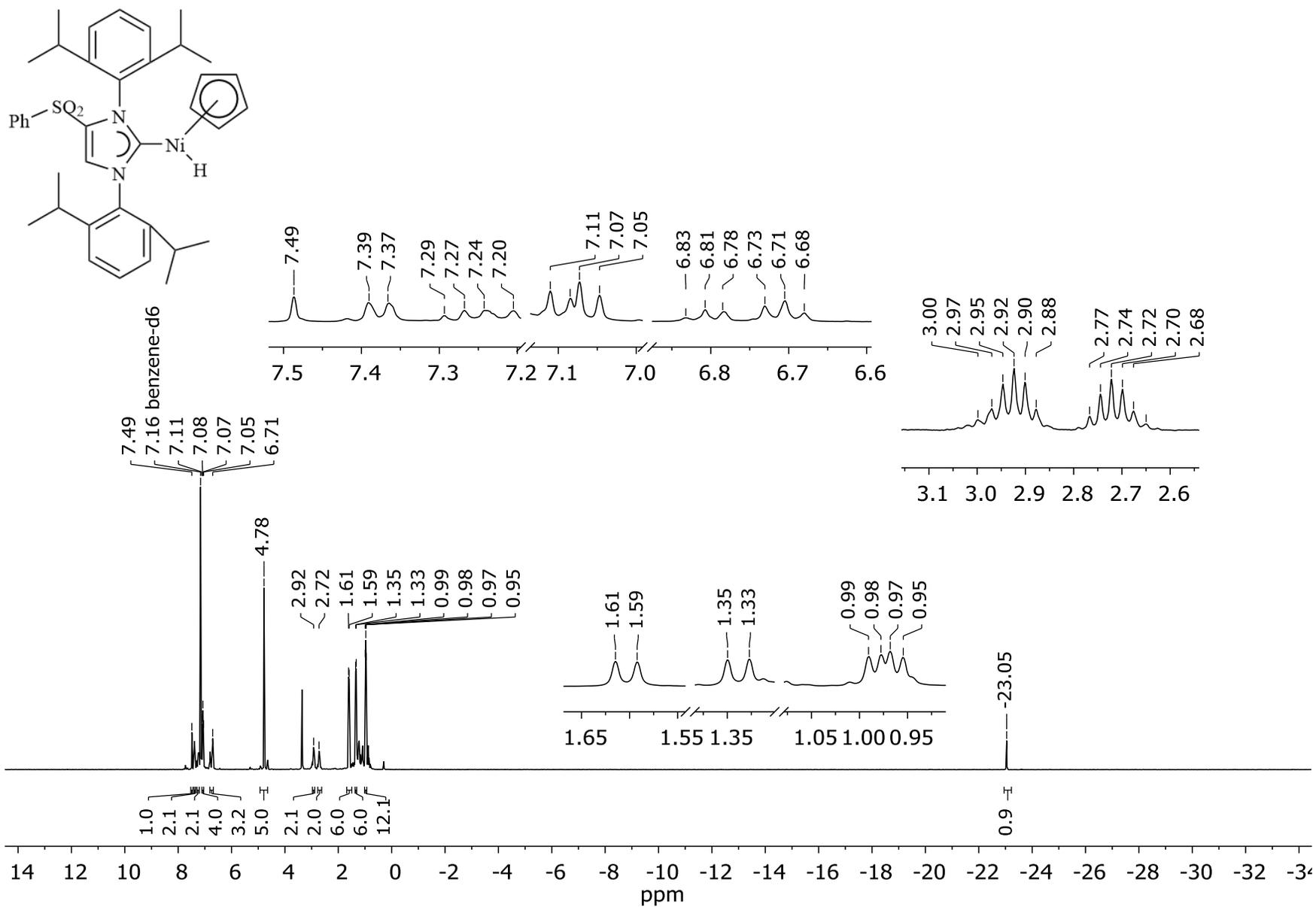
**Figure S7.** <sup>13</sup>C NMR spectrum of compound **2c** (C<sub>6</sub>D<sub>6</sub>, 75 MHz)



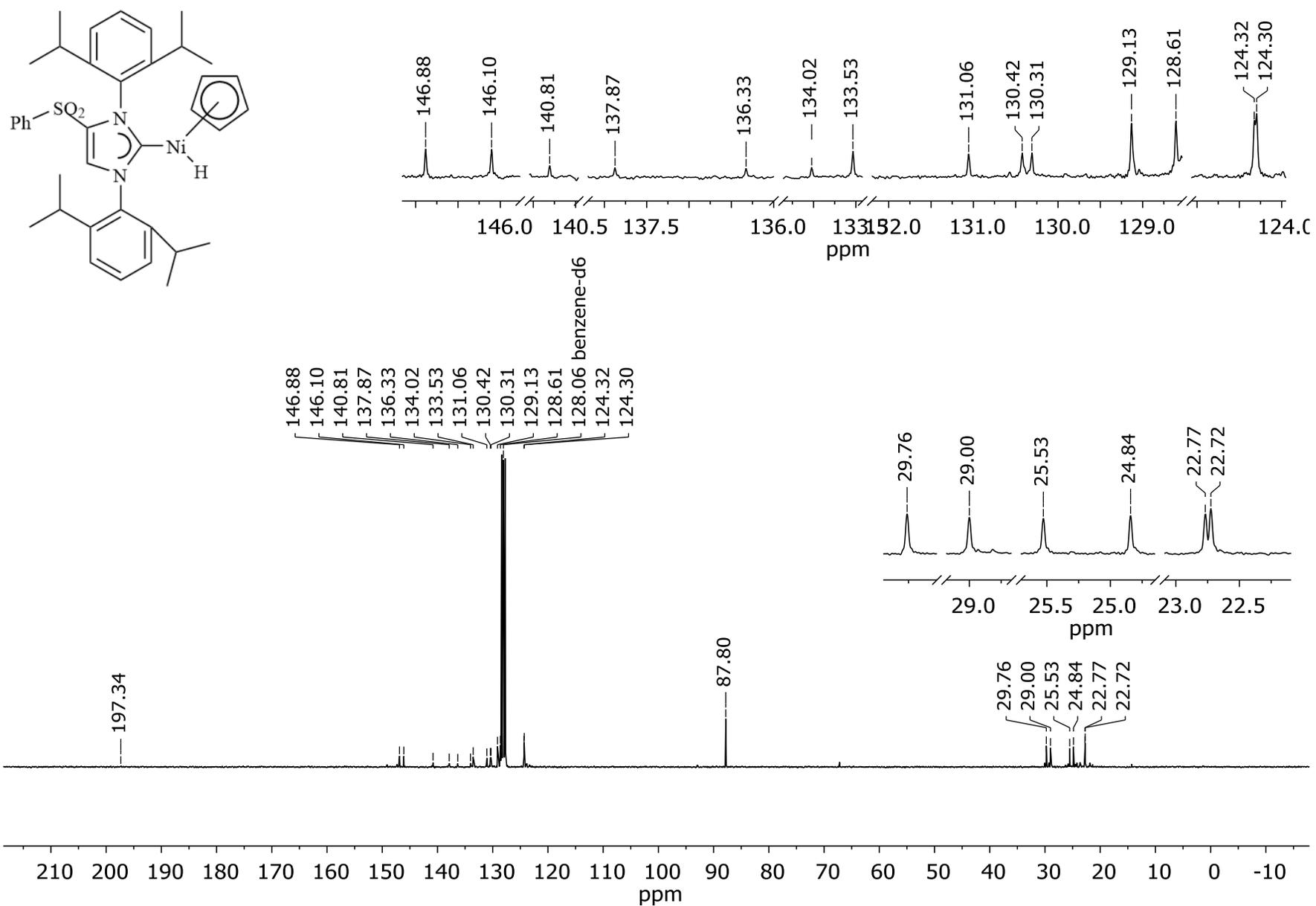
**Figure S8.**  $^1\text{H}$  NMR spectrum of compound **2d** ( $\text{C}_6\text{D}_6$ , 300 MHz)



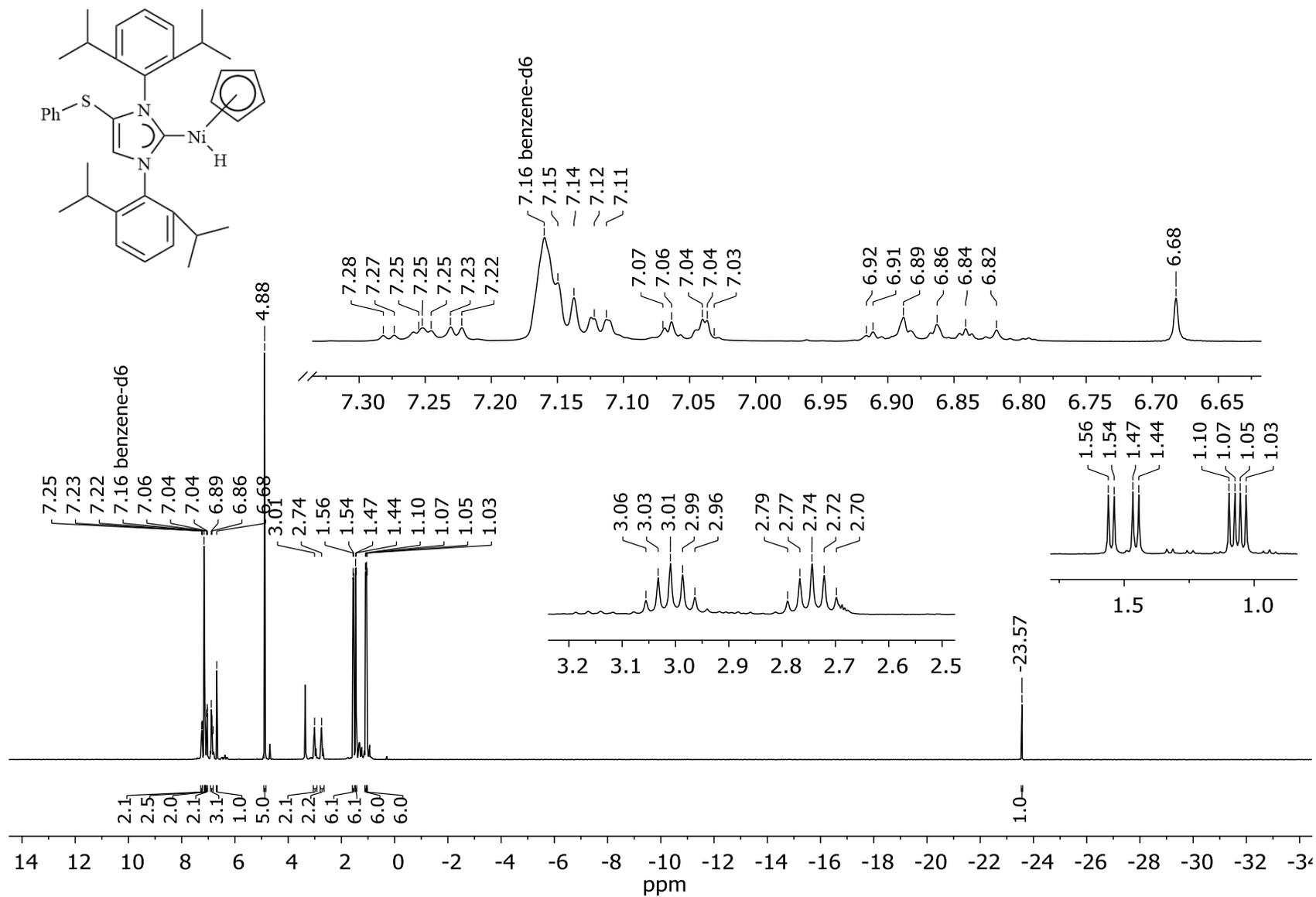
**Figure S9.**  $^{13}\text{C}$  NMR spectrum of compound **2d** ( $\text{C}_6\text{D}_6$ , 75 MHz)



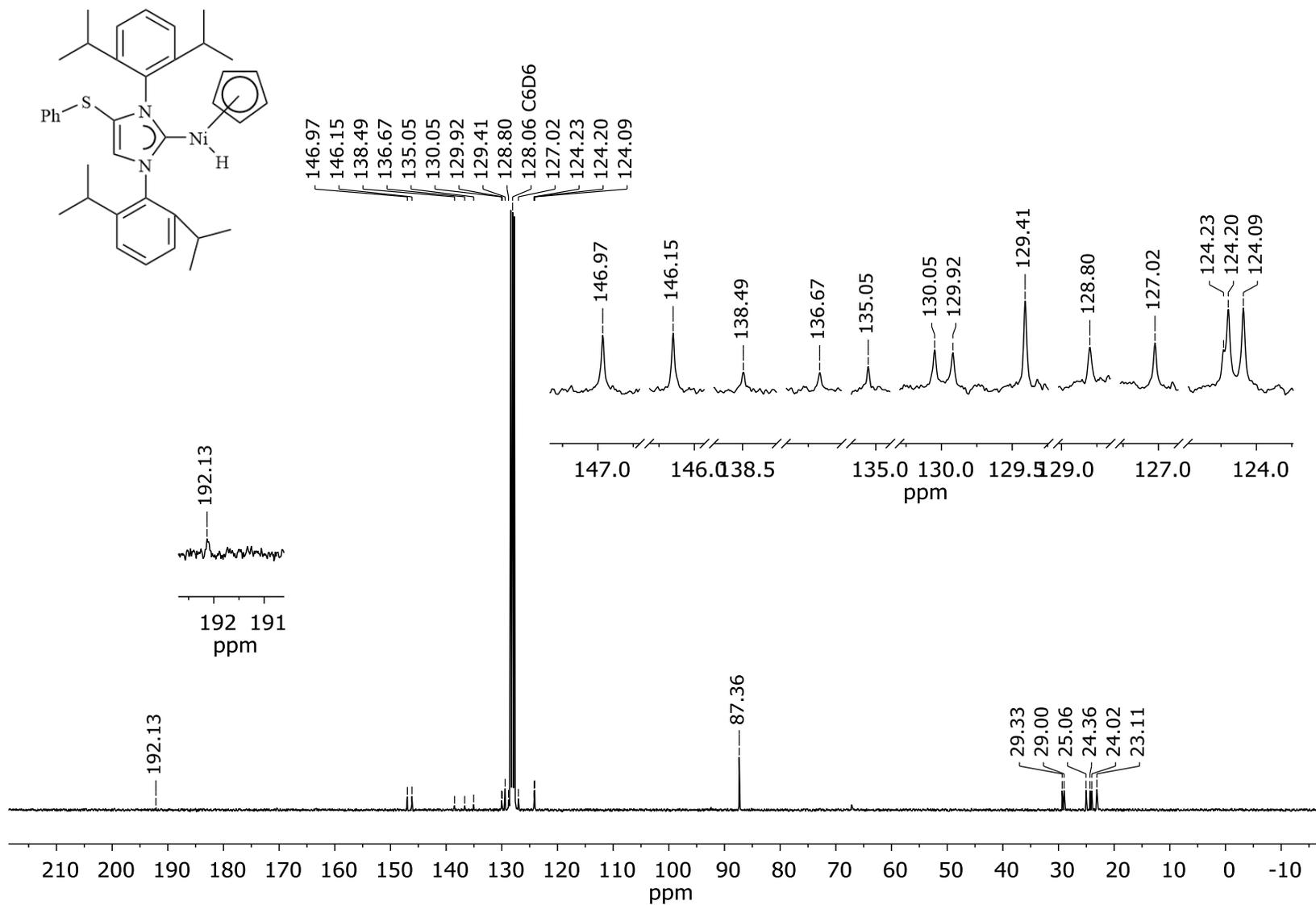
**Figure S10.** <sup>1</sup>H NMR spectrum of compound **2e** (C<sub>6</sub>D<sub>6</sub>, 300 MHz)



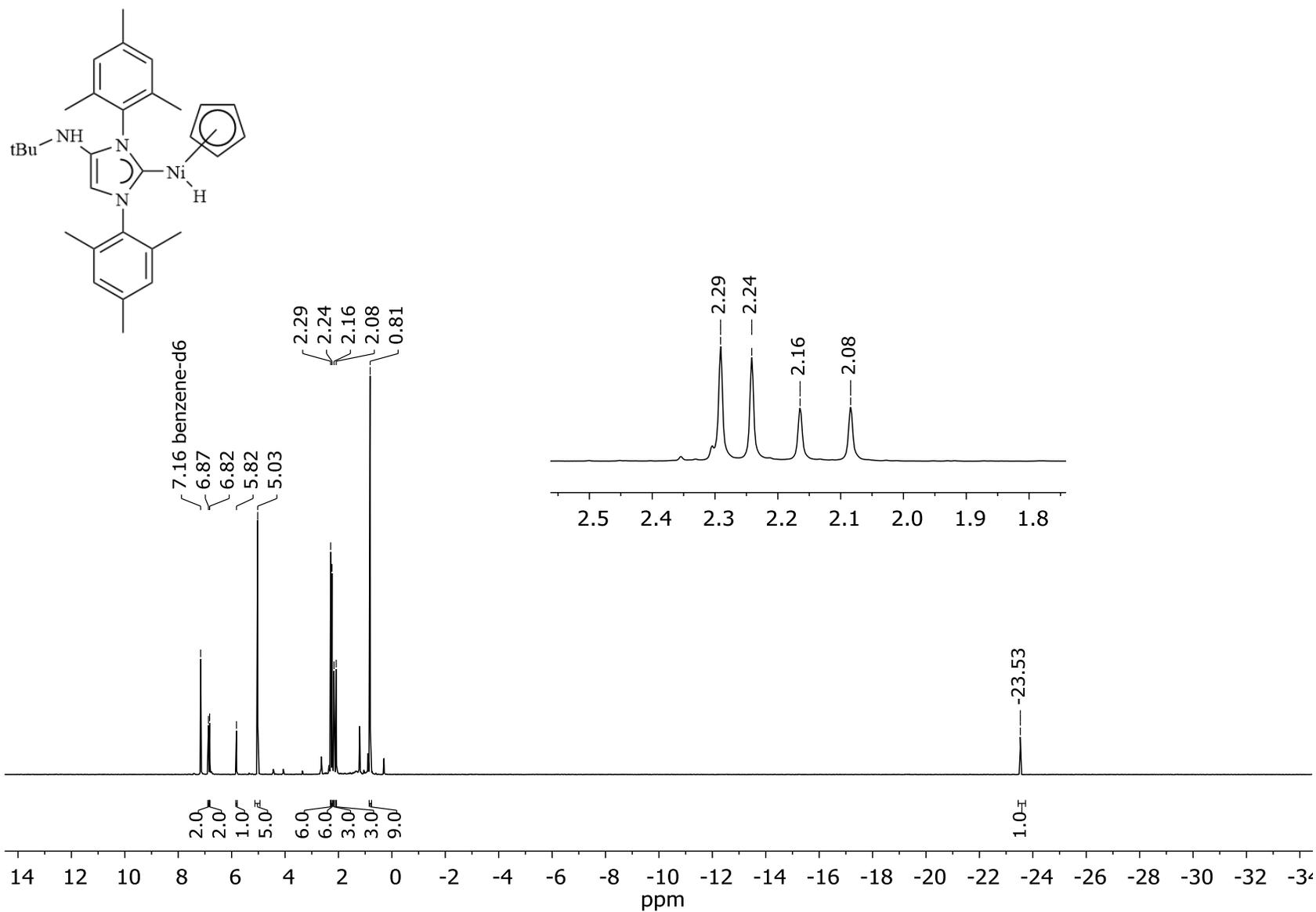
**Figure S11.**  $^{13}\text{C}$  NMR spectrum of compound **2e** ( $\text{C}_6\text{D}_6$ , 75 MHz)



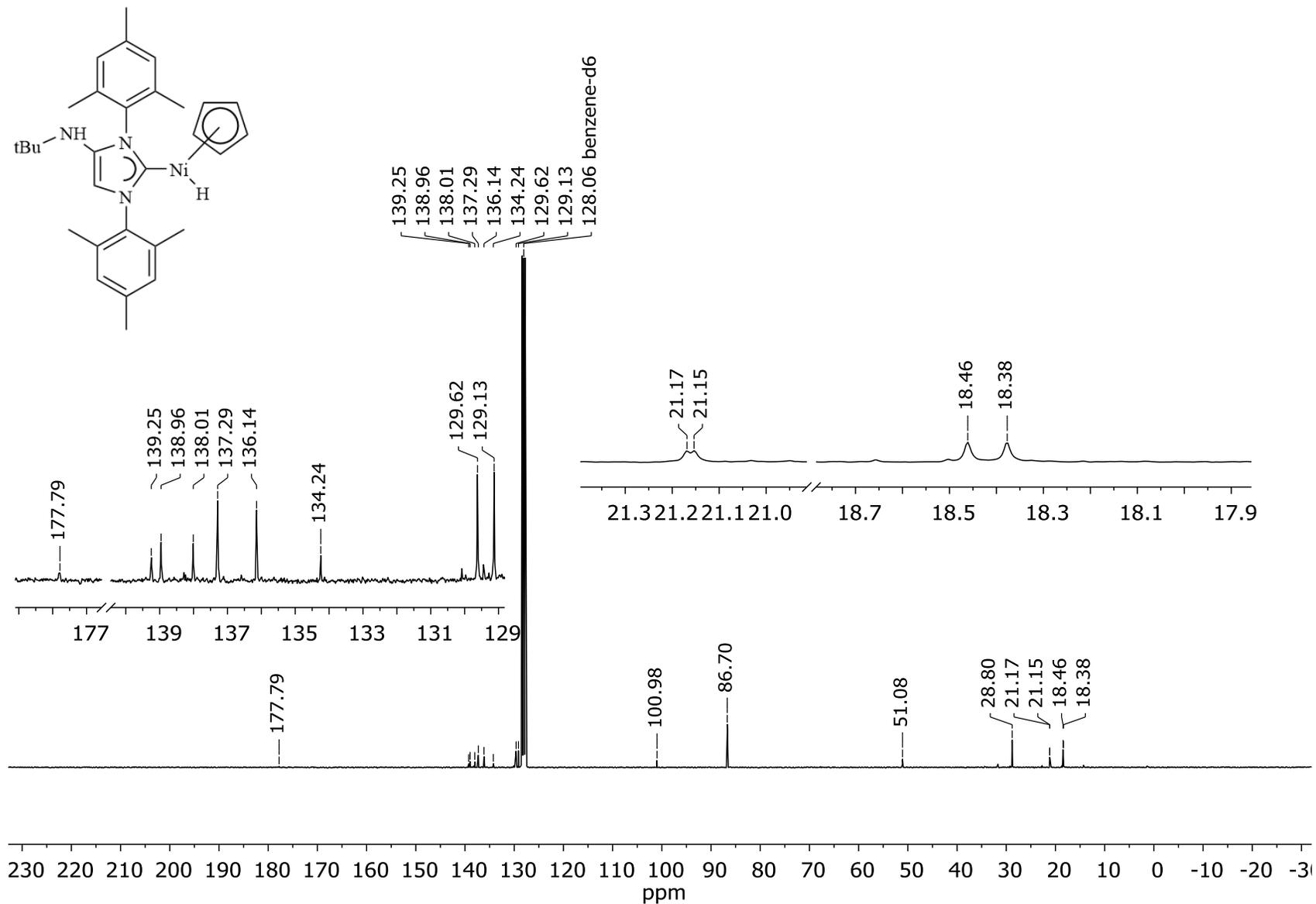
**Figure S12.**  $^1\text{H}$  NMR spectrum of compound **2f** ( $\text{C}_6\text{D}_6$ , 300 MHz)



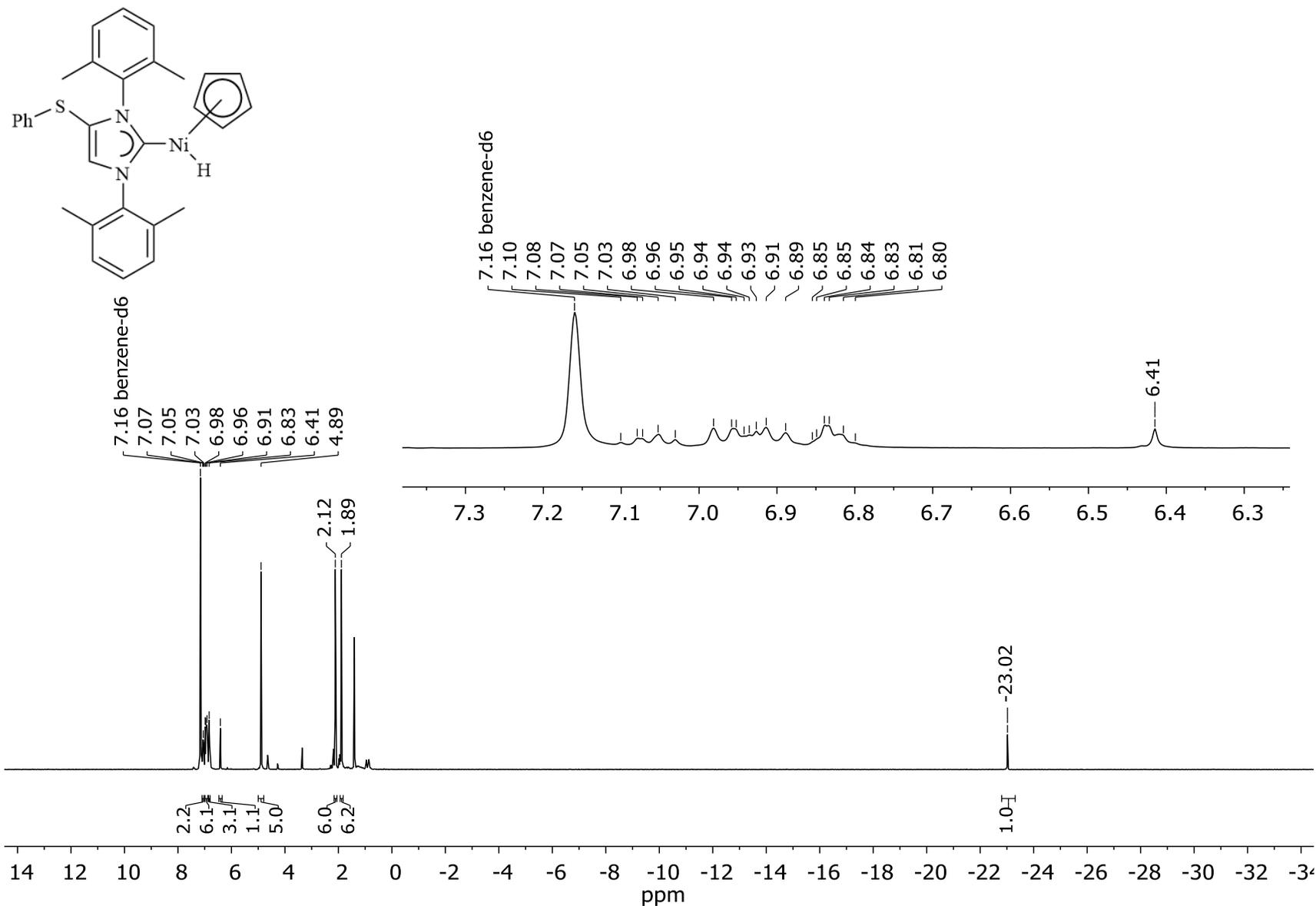
**Figure S13.**  $^{13}\text{C}$  NMR spectrum of compound **2f** ( $\text{C}_6\text{D}_6$ , 75 MHz)



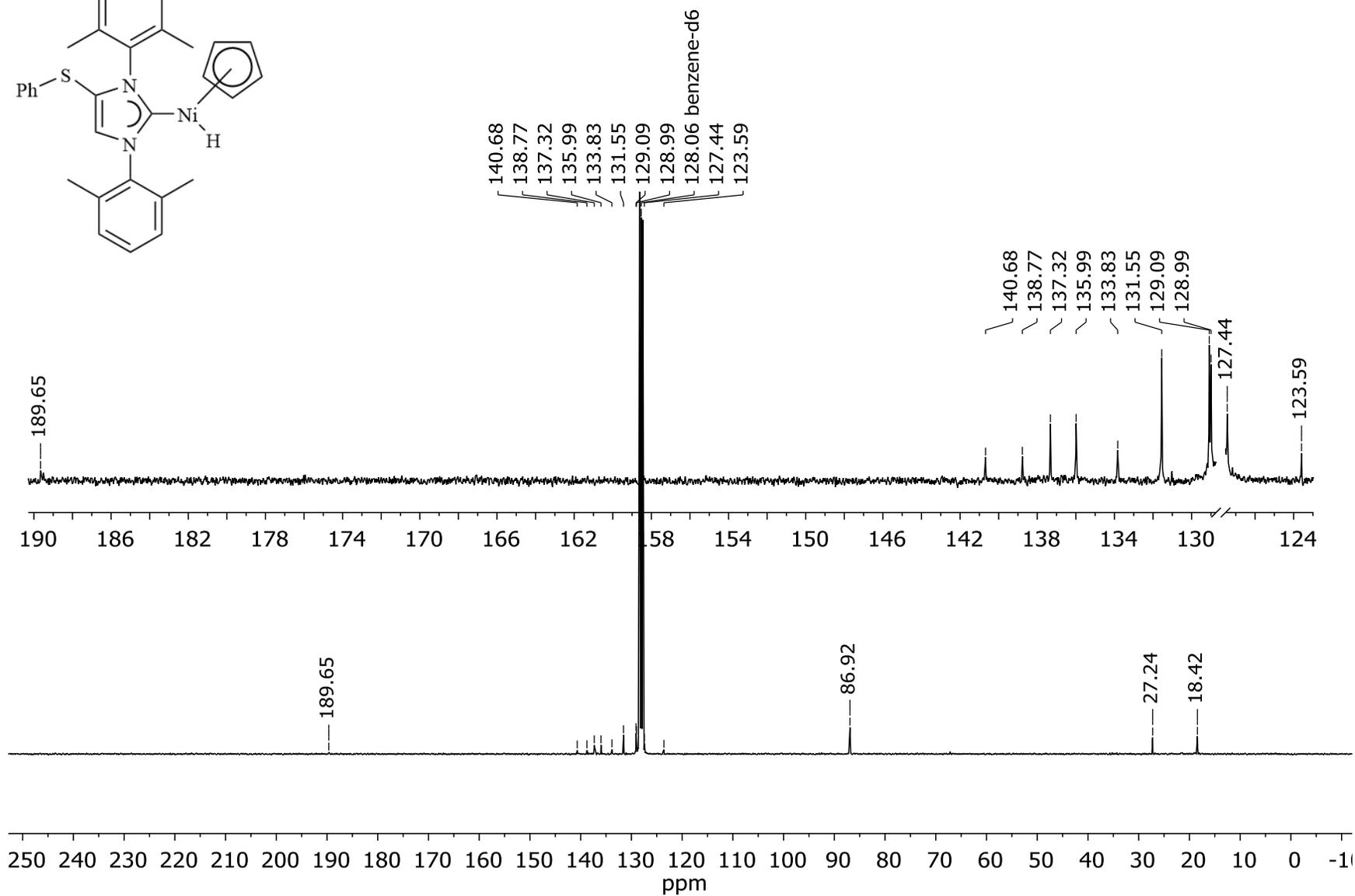
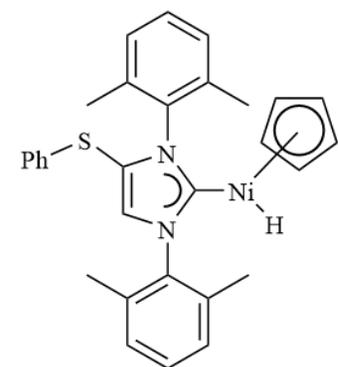
**Figure S14.**  $^1\text{H}$  NMR spectrum of compound **2g** ( $\text{C}_6\text{D}_6$ , 300 MHz)



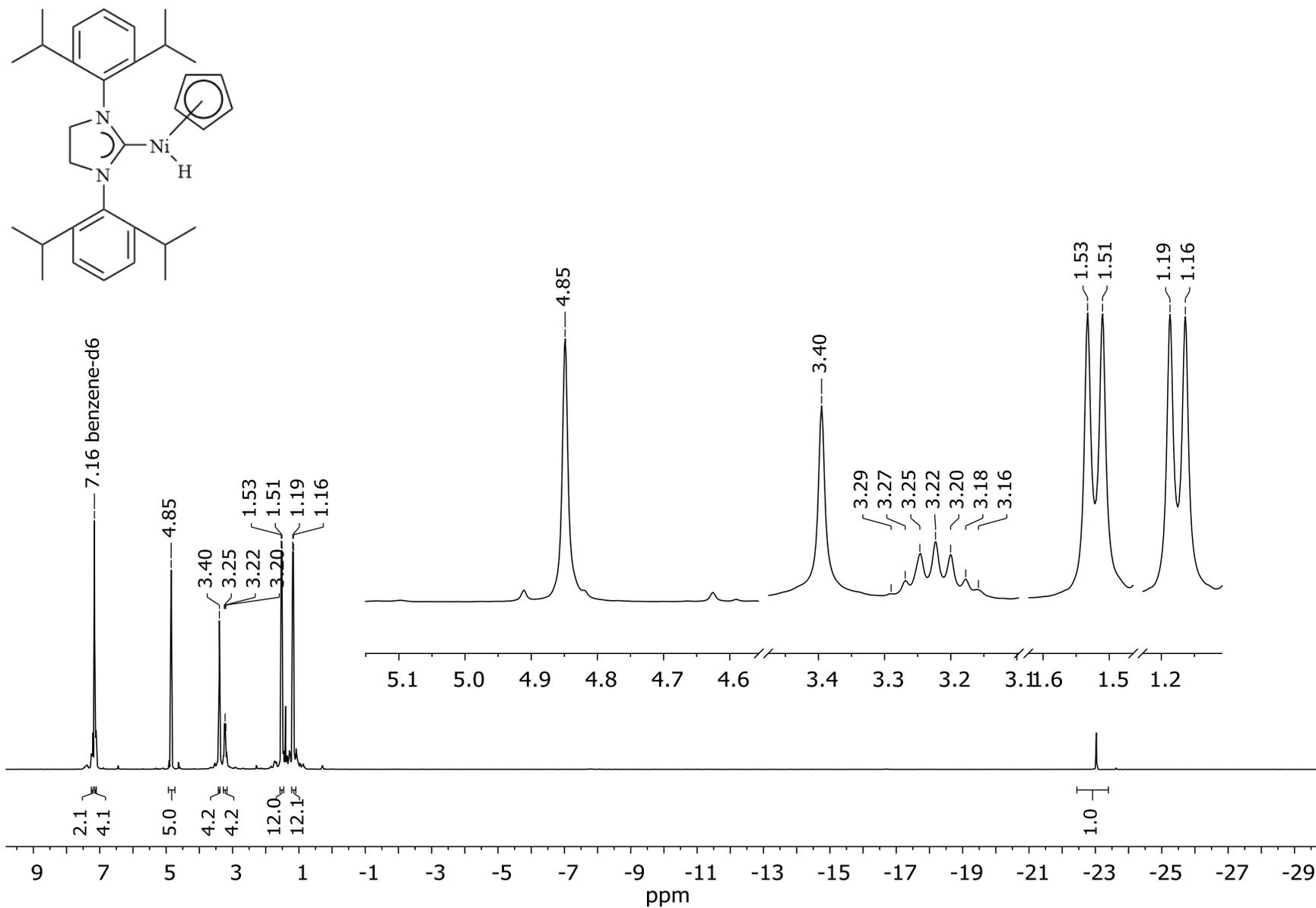
**Figure S15.**  $^{13}\text{C}$  NMR spectrum of compound **2g** ( $\text{C}_6\text{D}_6$ , 75 MHz)



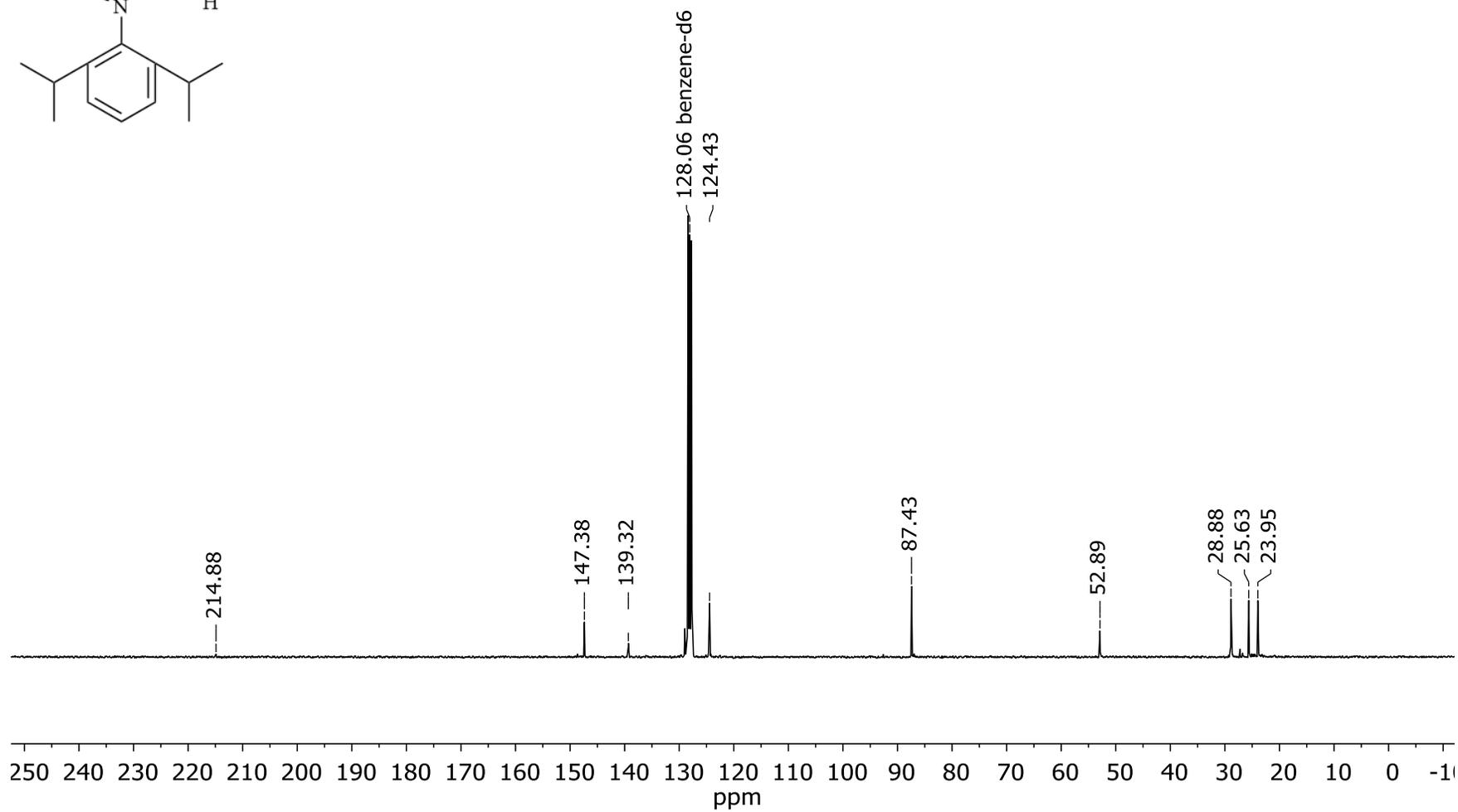
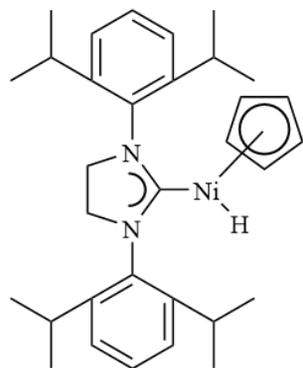
**Figure S16.**  $^1\text{H}$  NMR spectrum of compound **2h** ( $\text{C}_6\text{D}_6$ , 300 MHz)



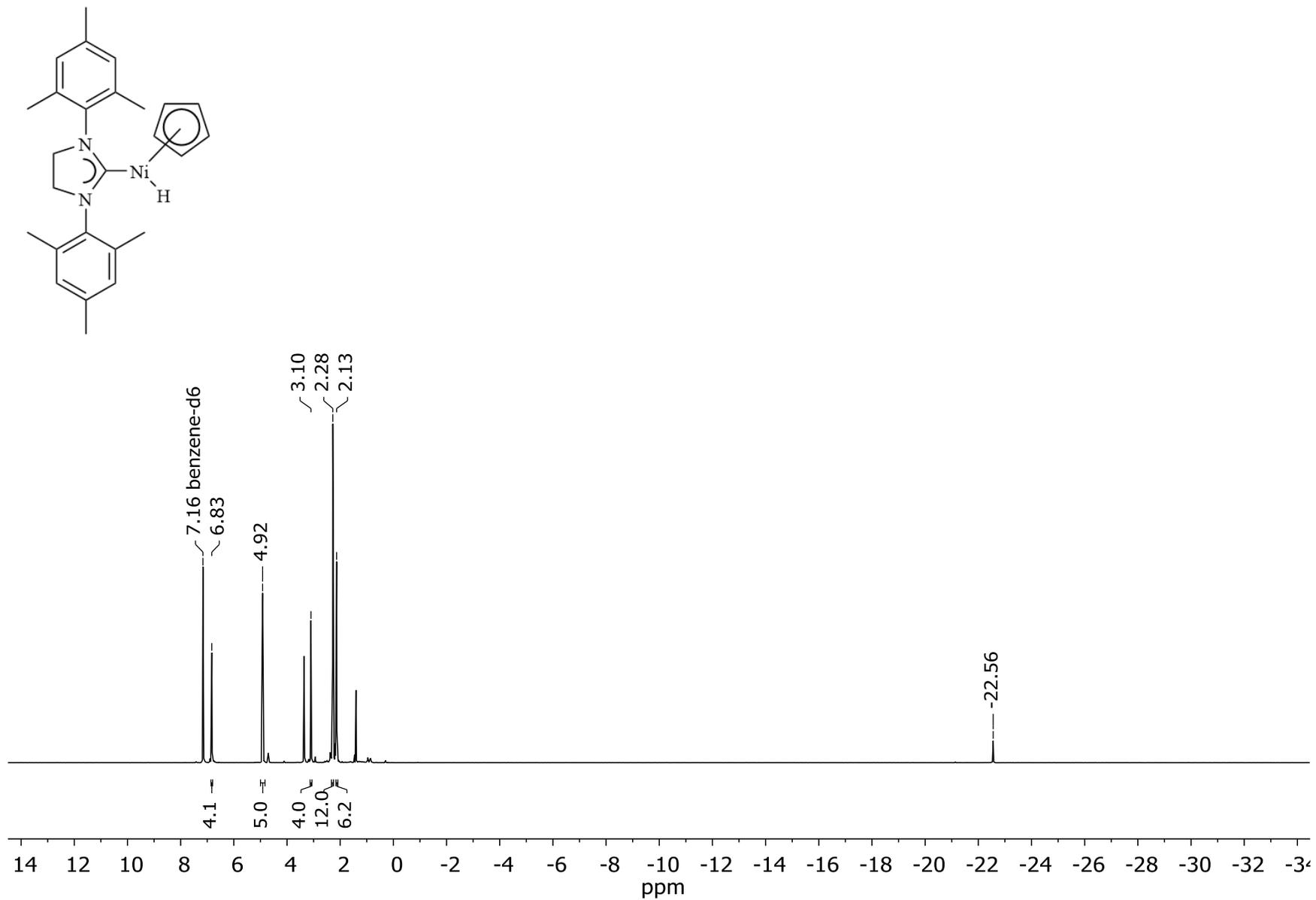
**Figure S17.** <sup>13</sup>C NMR spectrum of compound **2h** (C<sub>6</sub>D<sub>6</sub>, 75 MHz)



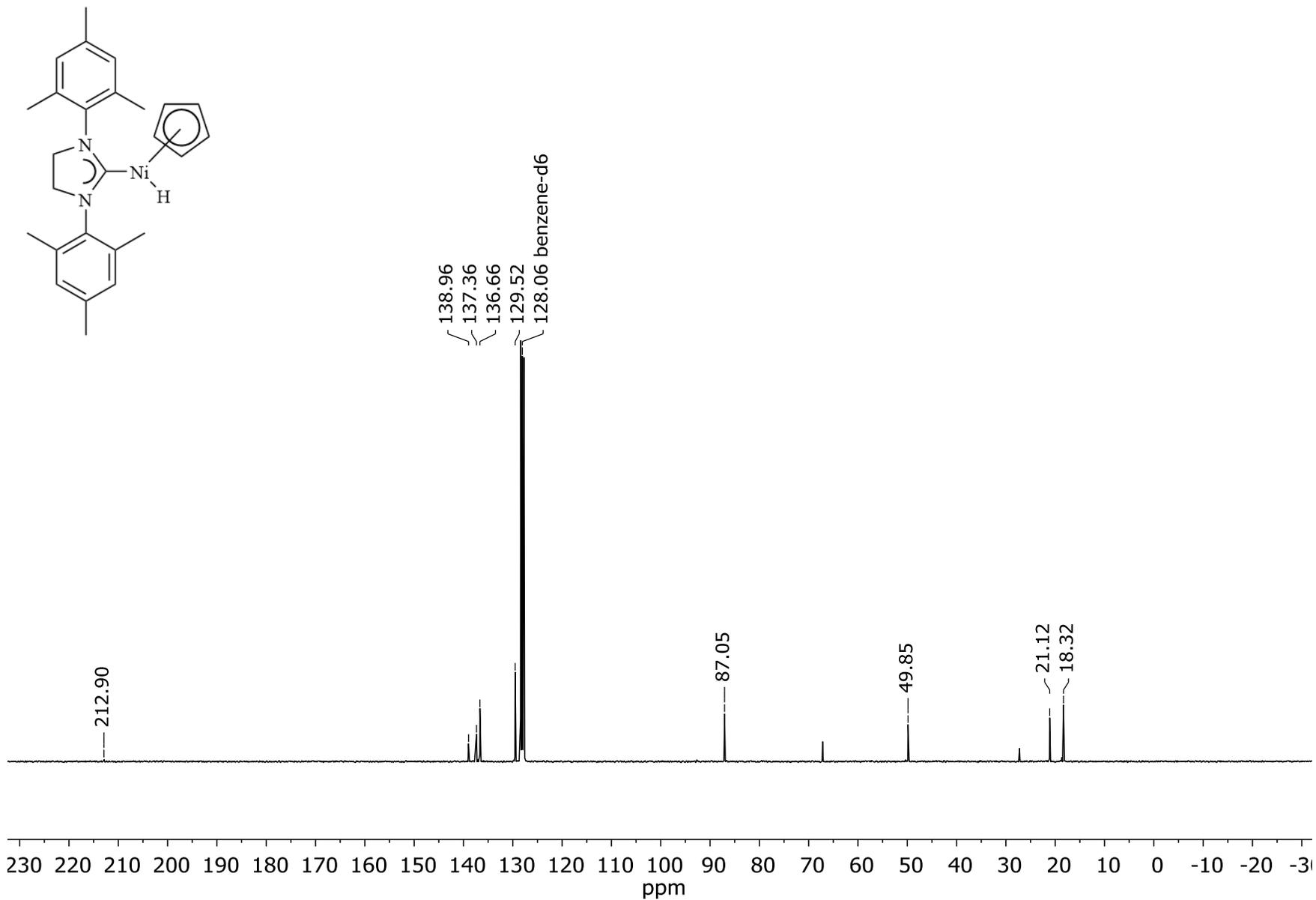
**Figure S18.**  $^1\text{H}$  NMR spectrum of compound **2i** ( $\text{C}_6\text{D}_6$ , 300 MHz)



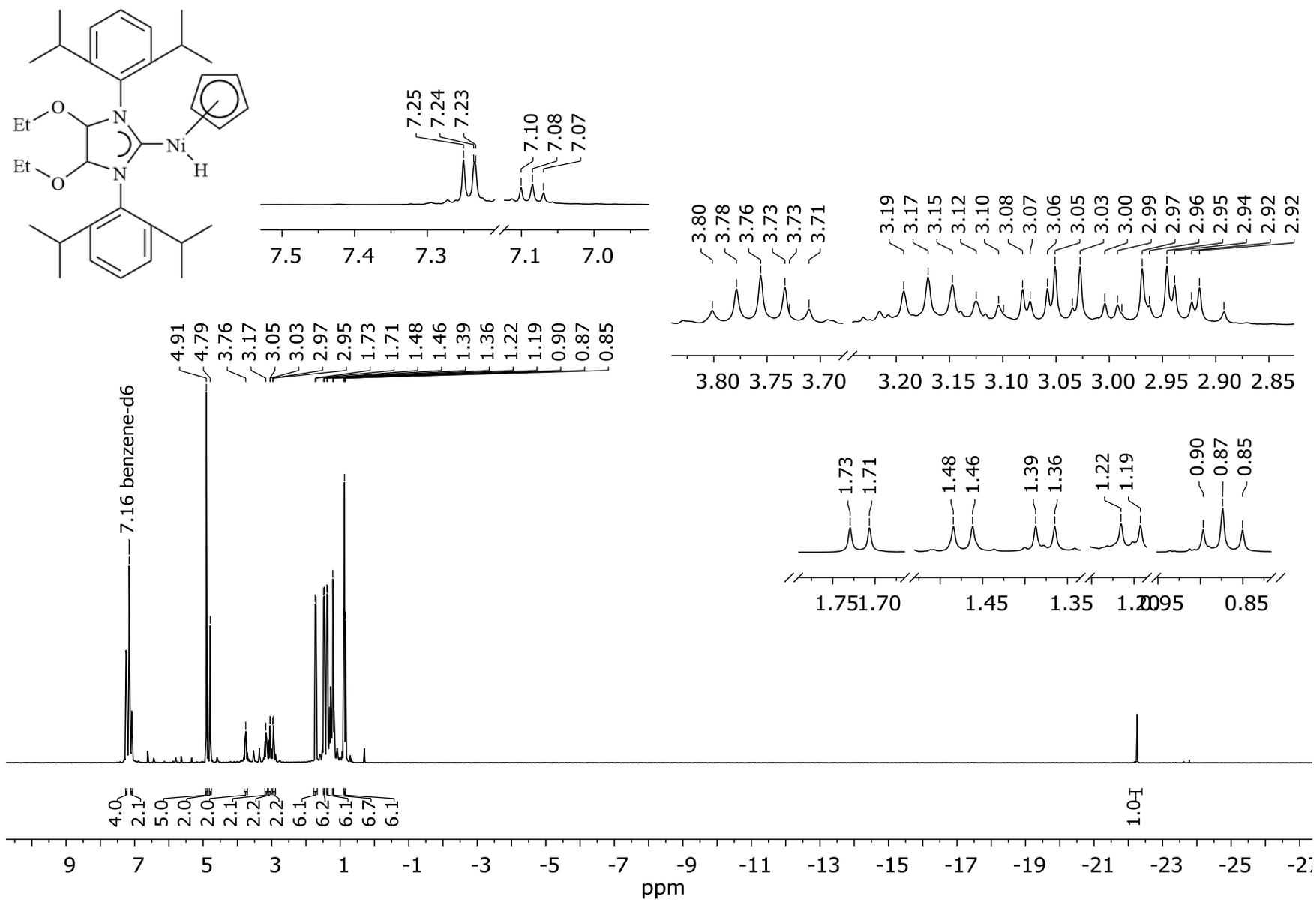
**Figure S19.** <sup>13</sup>C NMR spectrum of compound **2i** (C<sub>6</sub>D<sub>6</sub>, 75 MHz)



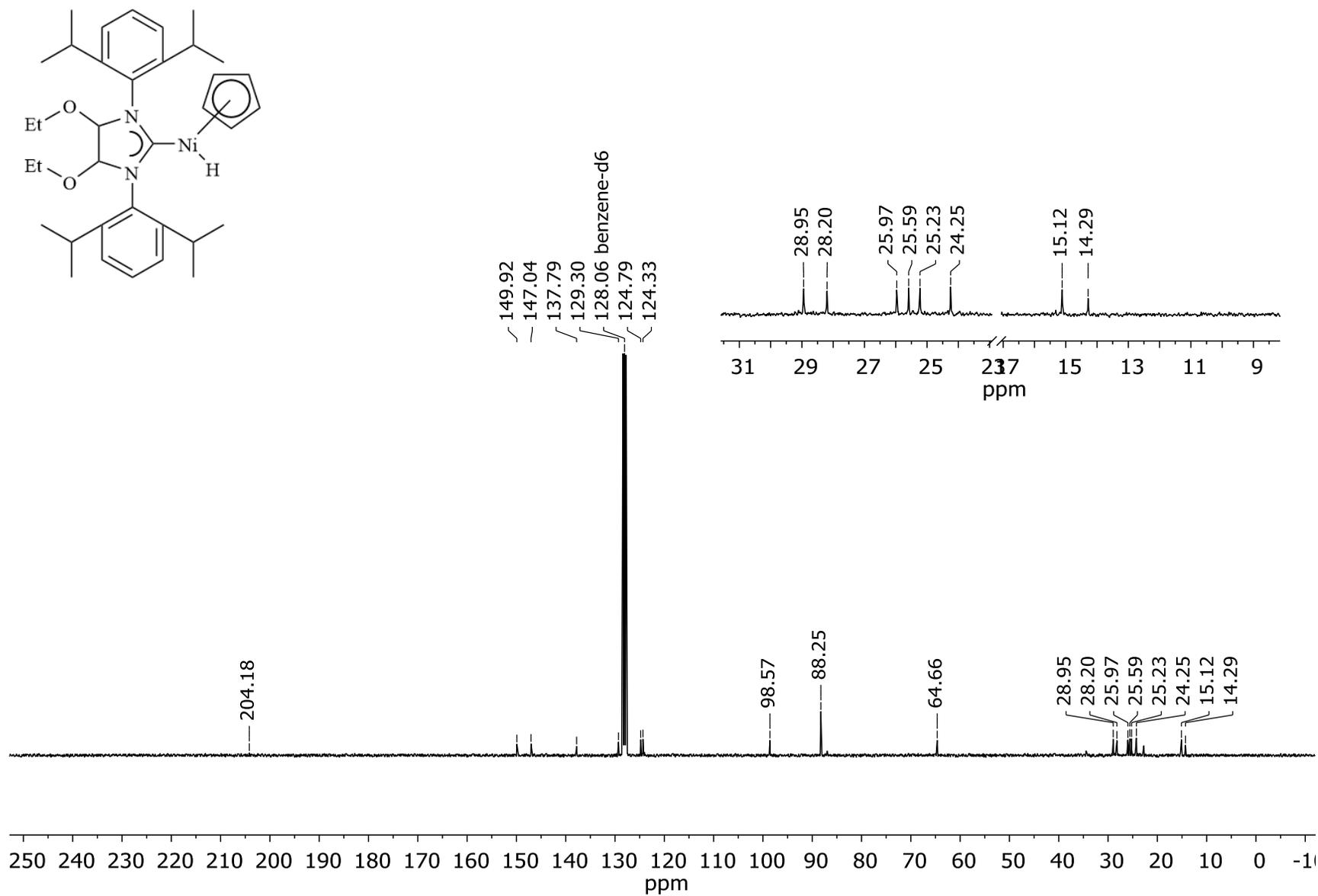
**Figure S20.**  $^1\text{H}$  NMR spectrum of compound **2j** ( $\text{C}_6\text{D}_6$ , 300 MHz)



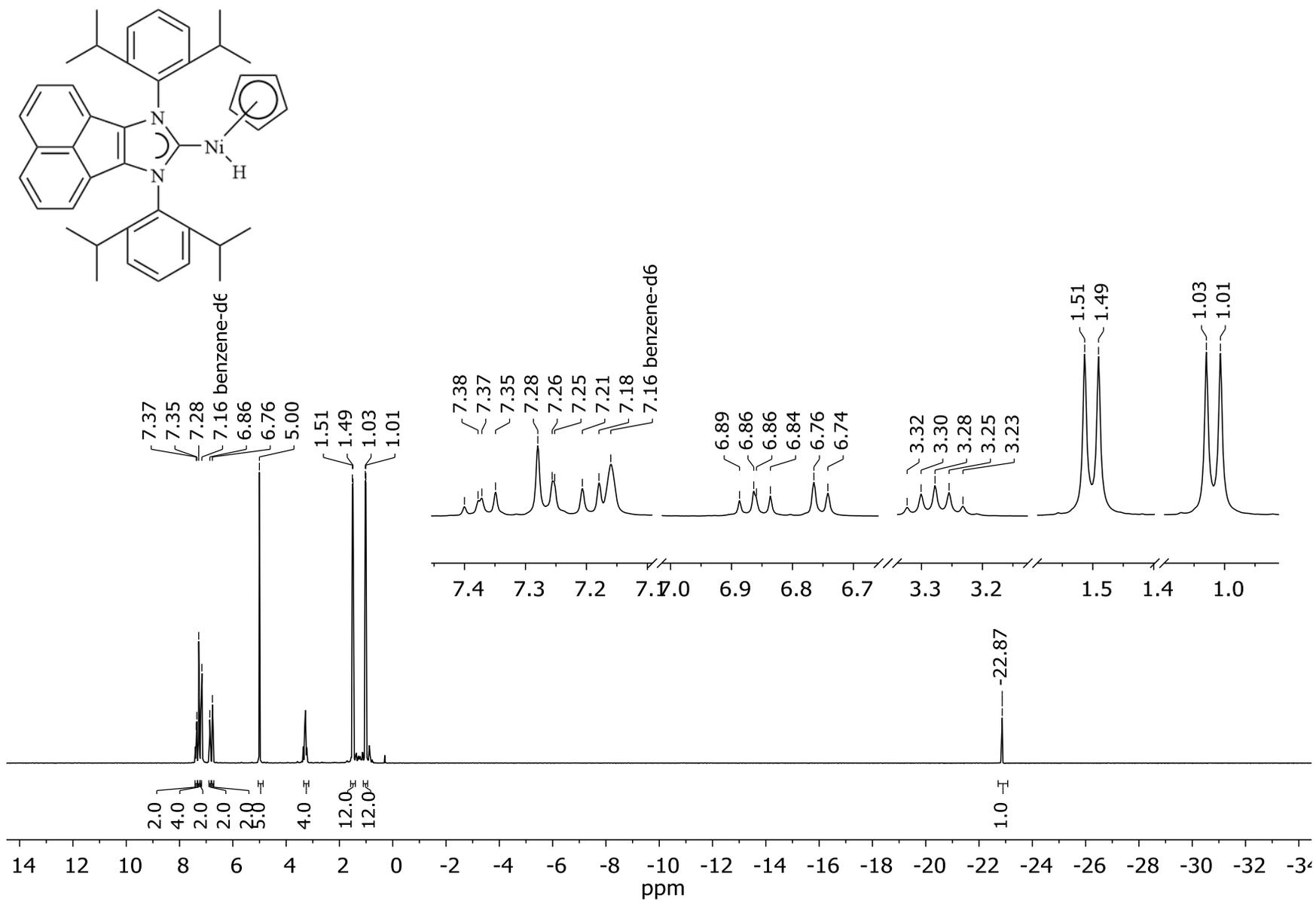
**Figure S21.**  $^{13}\text{C}$  NMR spectrum of compound **2j** ( $\text{C}_6\text{D}_6$ , 75 MHz)



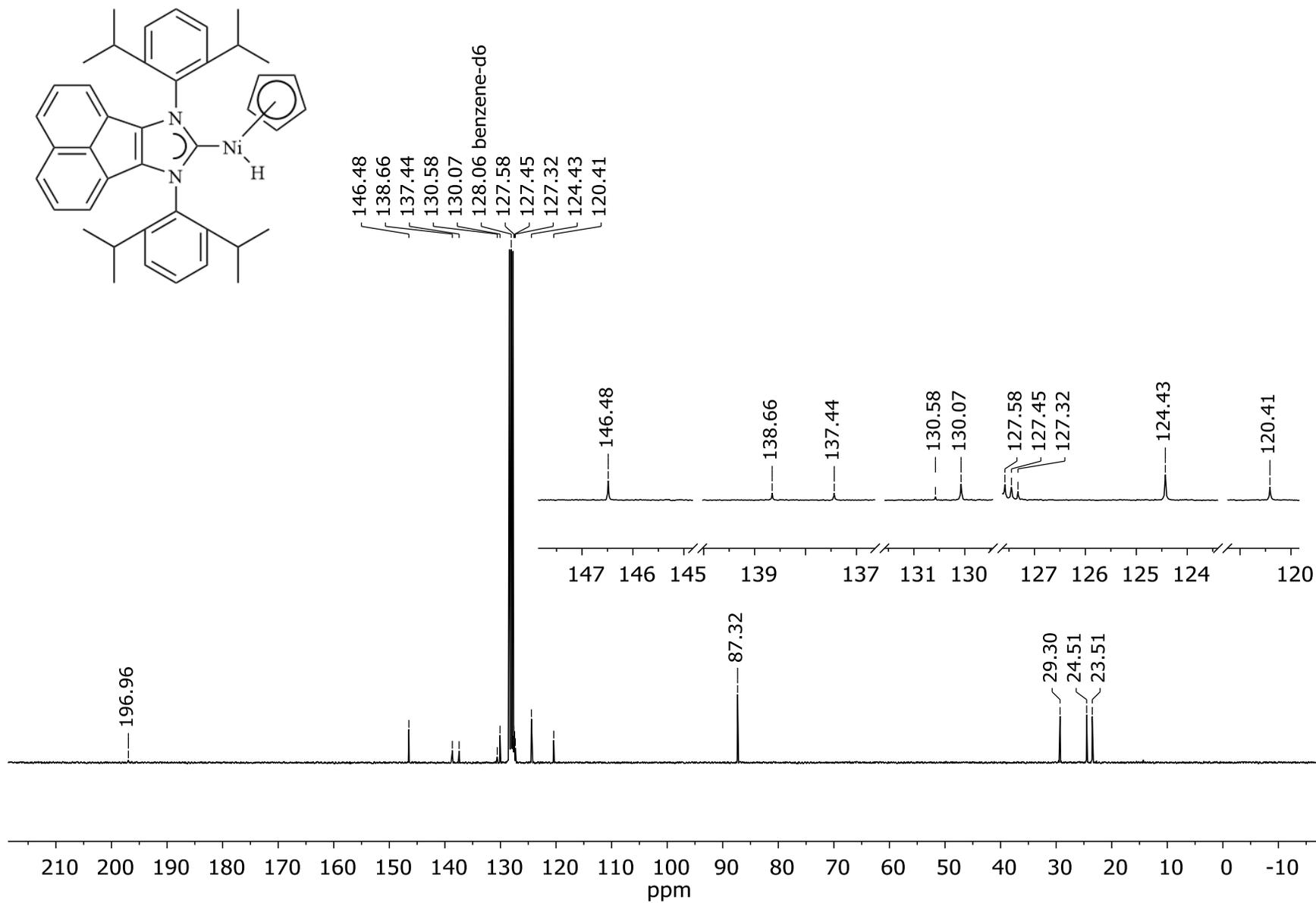
**Figure S22.**  $^1\text{H}$  NMR spectrum of compound **2k** ( $\text{C}_6\text{D}_6$ , 300 MHz)



**Figure S23.** <sup>13</sup>C NMR spectrum of compound **2k** (C<sub>6</sub>D<sub>6</sub>, 75 MHz)



**Figure S24.**  $^1\text{H}$  NMR spectrum of compound **2I** ( $\text{C}_6\text{D}_6$ , 300 MHz)



**Figure S25.**  $^{13}\text{C}$  NMR spectrum of compound **2l** ( $\text{C}_6\text{D}_6$ , 75 MHz)

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