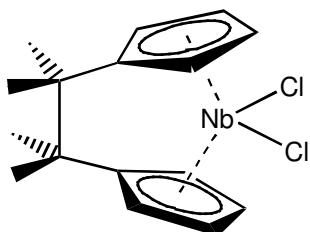


**Chapter 6**  
**Characterising Data**

**6.1 General**

NMR data are given as chemical shift ( $\delta$ ) [relative intensity, coupling, multiplicity, assignment] at 298 K unless otherwise stated. All IR data are expressed in wavenumbers ( $\text{cm}^{-1}$ ). For the FAB mass spectrometry data, the characteristic isotope patterns were observed.

**6.2 Characterising Data for Chapter 2****6.2.1 Characterising Data for  $[\text{Nb}\{(\eta\text{-C}_5\text{H}_4)\text{C}_2\text{Me}_4(\eta\text{-C}_5\text{H}_4)\}\text{Cl}_2]$  (1)****Appearance:**

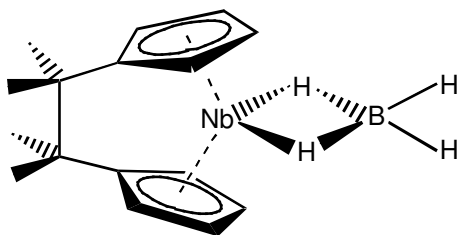
Brown solid

$$\text{C}_{16}\text{H}_{20}\text{Cl}_2\text{Nb} = 376.15$$

<b>Elemental Analyses:</b>	C %	H %	Cl %
Required:	51.1	5.4	18.9
Found:	50.8	5.1	19.4

<b>Mass Spectrometry:</b>	$m/z = 375$	$(\text{M})^+$	85 %
	$m/z = 340$	$(\text{M-Cl})^+$	17 %

**ESR:**  $\text{CH}_2\text{Cl}_2$ , 10 lines,  $g_{\text{iso}} = 2.008$ ,  $1a^{93}\text{Nb}|_{\text{iso}} = 110 \text{ G}$

6.2.2 Characterising Data for  $[\text{Nb}\{(\eta\text{-C}_5\text{H}_4)\text{C}_2\text{Me}_4(\eta\text{-C}_5\text{H}_4)\}(\eta^2\text{-BH}_4)]$  (2)**Appearance:**

Green solid

 $\text{C}_{16}\text{H}_{24}\text{BNb} = 320.08$ 

X-ray crystal structure: Appendix A

<b>Elemental Analyses:</b>	C %	H %	B %
Required:	60.0	7.6	3.4
Found:	60.1	7.7	3.3

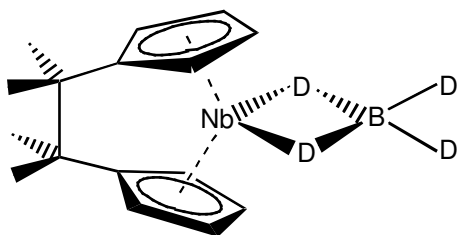
**$^1\text{H}$  NMR:**  $\text{C}_6\text{D}_5\text{CD}_3$  500 MHz  
 5.66 (m., 4H,  $\text{C}_5\text{H}_4$ )  
 5.52 (m., 4H,  $\text{C}_5\text{H}_4$ )  
 0.36 (m., 12H,  $\text{C}_2\text{Me}_4$ )

**$^{13}\text{C}\{^1\text{H}\}$  NMR:**  $\text{C}_6\text{D}_5\text{CD}_3$  125 MHz  
 103.11 (s.,  $\text{C}_5\text{H}_4$ )  
 85.23 (s.,  $\text{C}_5\text{H}_4$ )  
 43.79 (s.,  $\text{C}_2\text{Me}_4$ )  
 26.38 (s.,  $\text{C}_2\text{Me}_4$ )

**$^{11}\text{B}$  NMR:**  $\text{C}_6\text{D}_5\text{CD}_3$  160 MHz  
 26.9 (qn.,  $^1J_{\text{BH}} = 86$  Hz,  $\text{BH}_4$ )

**Selected IR data:** Nujol

2465  $\nu(\text{B-H}_t)$ , 2427  $\nu(\text{B-H}_t)$ ,  
 1712  $\nu(\text{B-H}_b)$ , 1661  $\nu(\text{B-H}_b)$

6.2.3 Characterising Data for  $[\text{Nb}\{(\eta\text{-C}_5\text{H}_4)\text{C}_2\text{Me}_4(\eta\text{-C}_5\text{H}_4)\}(\eta^2\text{-BD}_4)]$  (3)**Appearance:**

Green solid

$$\text{C}_{16}\text{H}_{22}\text{BD}_4\text{Nb} = 324.11$$

Elemental Analyses:	C %	H/D %	B %
Required:	59.3	8.8	3.3
Found:	59.1	8.8	3.3

**<sup>1</sup>H NMR:** C<sub>6</sub>D<sub>5</sub>CD<sub>3</sub> 500 MHz  
 5.70 (m., 4H, C<sub>5</sub>H<sub>4</sub>)  
 5.59 (m., 4H, C<sub>5</sub>H<sub>4</sub>)  
 0.37 (m., 12H, C<sub>2</sub>Me<sub>4</sub>)

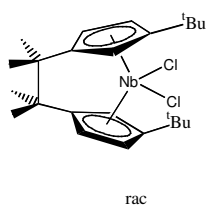
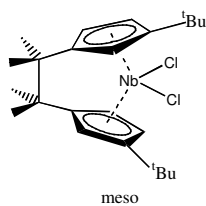
**<sup>13</sup>C{<sup>1</sup>H} NMR:** C<sub>6</sub>D<sub>5</sub>CD<sub>3</sub> 125 MHz  
 104.02 (s., C<sub>5</sub>H<sub>4</sub>)  
 85.72 (s., C<sub>5</sub>H<sub>4</sub>)  
 44.13 (s., C<sub>2</sub>Me<sub>4</sub>)  
 26.79 (s., C<sub>2</sub>Me<sub>4</sub>)

**<sup>2</sup>H NMR:** C<sub>6</sub>H<sub>5</sub>CH<sub>3</sub> 76.75 MHz  
 -4.91 (br. s, BD<sub>4</sub>)

**<sup>11</sup>B NMR:** C<sub>6</sub>D<sub>5</sub>CD<sub>3</sub> 160 MHz  
 25.5 (br. s, BD<sub>4</sub>)

**Selected IR data:** Nujol

1880 v(B-D<sub>t</sub>), 1769 v(B-D<sub>t</sub>),  
 1261 v(B-D<sub>b</sub>)

6.2.4 Characterising Data for  $[\text{Nb}\{(\eta\text{-C}_5\text{H}_3\text{-}^t\text{Bu})\text{C}_2\text{Me}_4(\eta\text{-C}_5\text{H}_3\text{-}^t\text{Bu})\}\text{Cl}_2]$  (4)**Appearance:**

Brown solid

 $\text{C}_{16}\text{H}_{24}\text{Cl}_2\text{Nb} = 488.36$ **Elemental Analyses:** C %                      H %

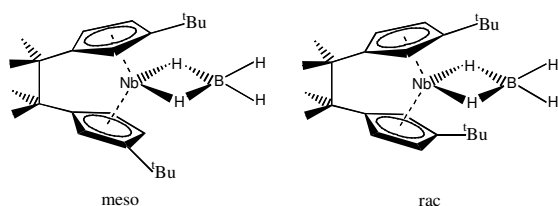
Required:                      59.0                      7.4

Found:                              58.4                      7.4

<b>Mass Spectrometry:</b>	$m/z = 487$	$(\text{M})^+$	85 %
	$m/z = 452$	$(\text{M-Cl})^+$	17 %

**ESR:**  $\text{CH}_2\text{Cl}_2$ , 10 lines,  $g_{\text{iso}} = 1.987$ ,  $1a^{93}\text{Nb}|_{\text{iso}} = 112 \text{ G}$

### 6.2.5 Characterising Data for $[\text{Nb}\{(\eta\text{-C}_5\text{H}_3\text{-}^t\text{Bu})\text{C}_2\text{Me}_4(\eta\text{-C}_5\text{H}_3\text{-}^t\text{Bu})\}(\eta^2\text{-BH}_4)]$ (5)

**Appearance:**

Green solid

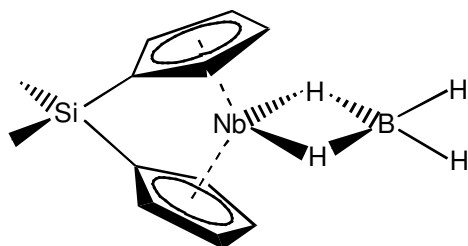
 $\text{C}_{16}\text{H}_{24}\text{BNb} = 432.30$ 

X-ray crystal structure: Appendix B

Elemental Analyses:	C %	H %	B %
Required:	66.7	9.3	2.5
Found:	66.0	9.5	2.4

$^1\text{H}$ NMR:	$\text{C}_6\text{D}_5\text{CD}_3$ 500 MHz	$^{13}\text{C}\{^1\text{H}\}$ NMR:	$\text{C}_6\text{D}_5\text{CD}_3$ 125 MHz
<i>rac</i> -	6.22 (s., 2H, $\text{C}_5\text{H}_3$ )	<i>rac</i> -	99.81 (s., $\text{C}_5\text{H}_3$ )
	5.37 (s., 2H, $\text{C}_5\text{H}_3$ )		84.70 (s., $\text{C}_5\text{H}_3$ )
	5.26 (s., 2H, $\text{C}_5\text{H}_3$ )		80.41 (s., $\text{C}_5\text{H}_3$ )
	1.40 (s., 18H, $^t\text{Bu}$ )		43.98 (s., $\underline{\text{C}}(\text{CH}_3)_3$ )
	0.53 (s., 6H, $\text{C}_2\text{Me}_4$ )		31.92 (s., $\text{C}(\underline{\text{C}}\text{H}_3)_3$ )
	0.49 (s., 6H, $\text{C}_2\text{Me}_4$ )		28.81 (s., $\underline{\text{C}}_2\text{Me}_4$ )
<i>meso</i> -	6.36 (s., 2H, $\text{C}_5\text{H}_3$ )	<i>meso</i> -	99.61 (s., $\text{C}_5\text{H}_3$ )
	5.13 (s., 2H, $\text{C}_5\text{H}_3$ )		87.08 (s., $\text{C}_5\text{H}_3$ )
	5.12 (s., 2H, $\text{C}_5\text{H}_3$ )		83.57 (s., $\text{C}_5\text{H}_3$ )
	1.31 (s., 18H, $^t\text{Bu}$ )		83.57 (s., $\text{C}_5\text{H}_3$ )
	0.48 (s., 6H, $\text{C}_2\text{Me}_4$ )		44.42 (s., $\underline{\text{C}}(\text{CH}_3)_3$ )
	0.40 (s., 6H, $\text{C}_2\text{Me}_4$ )		32.11 (s., $\text{C}(\underline{\text{C}}\text{H}_3)_3$ )
			27.12 (s., $\underline{\text{C}}_2\text{Me}_4$ )
			21.04 (s., $\underline{\text{C}}_2\text{Me}_4$ )

**Selected IR data:** Nujol2467  $\nu(\text{B-H}_t)$ , 2430  $\nu(\text{B-H}_t)$ ,1690  $\nu(\text{B-H}_b)$ , 1666  $\nu(\text{B-H}_b)$  $^{11}\text{B}$  NMR:  $\text{C}_6\text{D}_5\text{CD}_3$  160 MHz26.9 (qn.,  $^1J_{\text{BH}} = 86$  Hz,  $\text{BH}_4$ )

6.2.6 Characterising Data for  $[\text{Nb}\{(\eta\text{-C}_5\text{H}_4)\text{SiMe}_2(\eta\text{-C}_5\text{H}_4)\}(\eta^2\text{-BH}_4)]$  (6)**Appearance:**

Green solid

 $\text{C}_{12}\text{H}_{18}\text{BNbSi} = 294.08$ 

X-ray crystal structure: Appendix C

Elemental Analyses:	C %	H %	B %
Required:	49.0	6.2	3.7
Found:	49.3	6.2	3.4

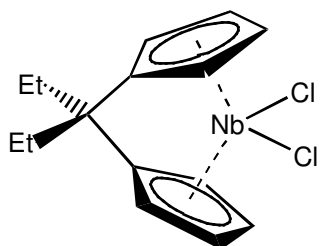
Mass Spectrometry:	m/z = 296	(M+2) <sup>+</sup>	45 %
	m/z = 279	(M-BH <sub>4</sub> ) <sup>+</sup>	26 %

<sup>1</sup> H NMR:	C <sub>6</sub> D <sub>5</sub> CD <sub>3</sub> 500 MHz	<sup>13</sup> C{ <sup>1</sup> H} NMR:	C <sub>6</sub> D <sub>5</sub> CD <sub>3</sub> 125 MHz
	6.03 (m., 4H, C <sub>5</sub> H <sub>4</sub> )		111.51 (s., C <sub>5</sub> H <sub>4</sub> )
	5.16 (m., 4H, C <sub>5</sub> H <sub>4</sub> )		87.14 (s., C <sub>5</sub> H <sub>4</sub> )
	-0.62 (s., 6H, SiMe <sub>2</sub> )		74.52 (s., C <sub>ipso</sub> C <sub>5</sub> H <sub>4</sub> )
			-6.78 (s., SiMe <sub>2</sub> )

<sup>11</sup> B NMR:	C <sub>6</sub> D <sub>5</sub> CD <sub>3</sub> 160 MHz
	26.8 (qn., <sup>1</sup> J <sub>BH</sub> = 86 Hz, BH <sub>4</sub> )

**Selected IR data:** Nujol

2460 ν(B-H<sub>t</sub>), 2410 ν(B-H<sub>t</sub>),  
1703 ν(B-H<sub>b</sub>), 1652 ν(B-H<sub>b</sub>)

**6.2.7 Characterising Data for [Nb{(η-C<sub>5</sub>H<sub>4</sub>)CEt<sub>2</sub>(η-C<sub>5</sub>H<sub>4</sub>)Cl<sub>2</sub>] (7)****Appearance:**

Purple solid

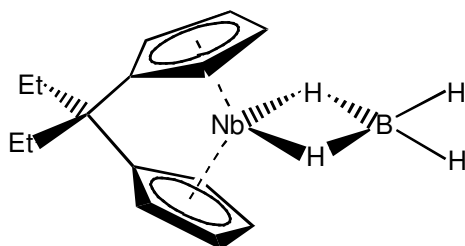
 $C_{15}H_{18}Cl_2Nb = 362.12$ **Elemental Analyses: C %                      H %**

Required:                      49.8                      5.0

Found:                              49.7                      5.3

<b>Mass Spectrometry:</b>	$m/z = 361$	$(M)^+$	85 %
	$m/z = 326$	$(M-Cl)^+$	17 %

**ESR:** CH<sub>2</sub>Cl<sub>2</sub>, 10 lines,  $g_{iso} = 1.998$ ,  $la^{93}Nb|_{iso} = 94$  G

6.2.8 Characterising Data for  $[\text{Nb}\{\eta\text{-C}_5\text{H}_4\}\text{CEt}_2\{\eta\text{-C}_5\text{H}_4\}\{\eta^2\text{-BH}_4\}]$  (8)**Appearance:**

Green solid

 $\text{C}_{15}\text{H}_{22}\text{BNb} = 306.06$ 

X-ray crystal structure: Appendix D

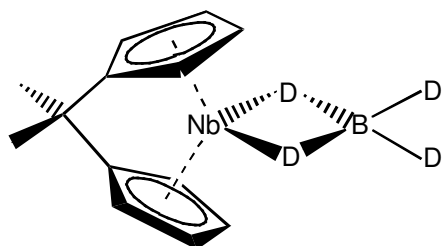
Elemental Analyses:	C %	H %	B %
Required:	58.9	7.3	3.5
Found:	57.8	7.7	3.0

$^1\text{H}$ NMR:	$\text{C}_6\text{D}_5\text{CD}_3$ 500 MHz	$^{13}\text{C}\{^1\text{H}\}$ NMR:	$\text{C}_6\text{D}_5\text{CD}_3$ 125 MHz
	5.67 (m., 4H, $\text{C}_5\text{H}_4$ )		106.21 (s., $\text{C}_5\text{H}_4$ )
	4.59 (m., 4H, $\text{C}_5\text{H}_4$ )		79.73 (s., $\text{C}_5\text{H}_4$ )
	0.44 (t., 6H, $\text{CH}_2\text{CH}_3$ )		77.01 (s., $\text{C}_{ipso}$ $\text{C}_5\text{H}_4$ )
	0.31 (q., 4H, $\text{CH}_2\text{CH}_3$ )		20.68 (s., $\text{CEt}_2$ )
	-3.38 (br. s., 4H, $\text{BH}_4$ )		6.11 (s., $\text{CEt}_2$ )

$^{11}\text{B}$ NMR:	$\text{C}_6\text{D}_5\text{CD}_3$ 160 MHz
	20.5 (qn., $^1\text{J}_{\text{BH}} = 85$ Hz, $\text{BH}_4$ )

**Selected IR data:** Nujol

2442  $\nu(\text{B-H}_t)$ , 2395  $\nu(\text{B-H}_t)$ ,  
 1715  $\nu(\text{B-H}_b)$ , 1703  $\nu(\text{B-H}_b)$

6.2.9 Characterising Data for  $[\text{Nb}\{\{\eta\text{-C}_5\text{H}_4\}\text{CMe}_2\{\eta\text{-C}_5\text{H}_4\}\}(\eta^2\text{-BD}_4)]$  (9)**Appearance:**

Green solid

 $\text{C}_{13}\text{H}_{14}\text{BD}_4\text{Nb} = 282.03$ 

Elemental Analyses:	C %	H/D %	B %
Required:	55.4	7.9	3.8
Found:	55.0	8.0	4.1

**$^1\text{H}$  NMR:**  $\text{C}_6\text{D}_5\text{CD}_3$  500 MHz  
 5.65 (m., 4H,  $\text{C}_5\text{H}_4$ )  
 4.58 (m., 4H,  $\text{C}_5\text{H}_4$ )  
 0.10 (s., 6H,  $\text{CMe}_3$ )

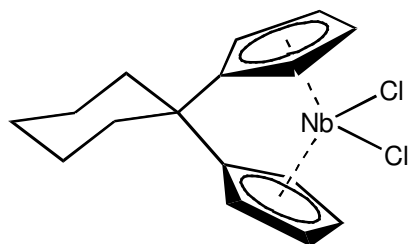
**$^{13}\text{C}\{^1\text{H}\}$  NMR:**  $\text{C}_6\text{D}_5\text{CD}_3$  125 MHz  
 106.31 (s.,  $\text{C}_5\text{H}_4$ )  
 79.48 (s.,  $\text{C}_5\text{H}_4$ )  
 73.17 (s.,  $\text{C}_{ipso}$   $\text{C}_5\text{H}_4$ )  
 20.92 (s.,  $\text{CMe}_2$ )

**$^2\text{H}$  NMR:**  $\text{C}_6\text{H}_5\text{CH}_3$  76.75 MHz  
 -2.99 (br. s,  $\text{BD}_4$ )

**$^{11}\text{B}$  NMR:**  $\text{C}_6\text{D}_5\text{CD}_3$  160 MHz  
 21.63 (br. s,  $\text{BD}_4$ )

**Selected IR data:** Nujol

1860  $\nu(\text{B-D}_t)$ , 1790  $\nu(\text{B-D}_t)$ ,  
 1270  $\nu(\text{B-D}_b)$

6.2.10 Characterising Data for  $[\text{Nb}\{(\eta\text{-C}_5\text{H}_4)\text{C}(\text{C}_5\text{H}_{10})(\eta\text{-C}_5\text{H}_4)\}\text{Cl}_2]$  (10)**Appearance:**

Brown solid

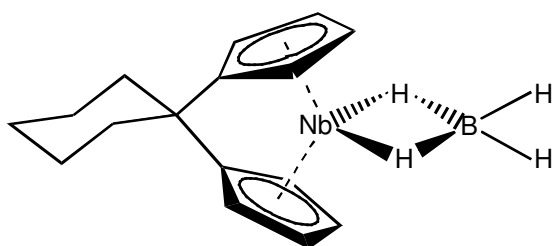
 $\text{C}_{16}\text{H}_{18}\text{Cl}_2\text{Nb} = 374.13$ 

<b>Elemental Analyses:</b>	<b>C %</b>	<b>H %</b>
Required:	51.4	4.9
Found:	51.7	5.5

<b>Mass Spectrometry:</b>	<b>m/z = 373</b>	<b>(M)<sup>+</sup></b>	<b>85 %</b>
	<b>m/z = 338</b>	<b>(M-Cl)<sup>+</sup></b>	<b>17 %</b>
	<b>m/z = 303</b>	<b>(M-2Cl)<sup>+</sup></b>	<b>25 %</b>

**ESR:**  $\text{CH}_2\text{Cl}_2$ , 10 lines,  $g_{\text{iso}} = 1.994$ ,  $1a^{93}\text{Nb}|_{\text{iso}} = 95 \text{ G}$

**6.2.11 Characterising Data for [Nb{(η-C<sub>5</sub>H<sub>4</sub>)C(C<sub>5</sub>H<sub>10</sub>)(η-C<sub>5</sub>H<sub>4</sub>)}  
(η<sup>2</sup>-BH<sub>4</sub>)] (11)**

**Appearance:**

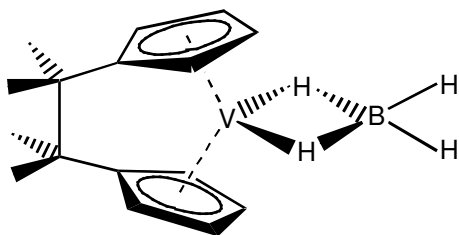
Green solid

C<sub>16</sub>H<sub>22</sub>BNb = 318.07

<b>Elemental Analyses:</b>		C %	H %
Required:		59.9	7.0
Found:		60.4	6.7

<b><sup>1</sup>H NMR:</b>	C <sub>6</sub> D <sub>5</sub> CD <sub>3</sub> 500 MHz	<b><sup>13</sup>C{<sup>1</sup>H} NMR:</b>	C <sub>6</sub> D <sub>5</sub> CD <sub>3</sub> 125 MHz
	5.70 (m., 4H, C <sub>5</sub> H <sub>4</sub> )		106.43 (s., C <sub>5</sub> H <sub>4</sub> )
	4.61 (m., 4H, C <sub>5</sub> H <sub>4</sub> )		81.31 (s., C <sub>ipso</sub> C <sub>5</sub> H <sub>4</sub> )
	1.11 (m., 4H, C(C <sub>5</sub> H <sub>10</sub> ))		79.49 (s., C <sub>5</sub> H <sub>4</sub> )
	0.38 (m., 4H, C(C <sub>5</sub> H <sub>10</sub> ))		37.88 (s., C( <u>C</u> <sub>5</sub> H <sub>10</sub> ))
	0.28 (m., 2H, C(C <sub>5</sub> H <sub>10</sub> ))		30.62 (s., C( <u>C</u> <sub>5</sub> H <sub>10</sub> ))
	-3.37 (br. s., 4H, BH <sub>4</sub> )		30.21 (s., C( <u>C</u> <sub>5</sub> H <sub>10</sub> ))
			26.20 (s., C( <u>C</u> <sub>5</sub> H <sub>10</sub> ))
<b><sup>11</sup>B NMR:</b>	C <sub>6</sub> D <sub>5</sub> CD <sub>3</sub> 160 MHz		
	18.2 (qn., <sup>1</sup> J <sub>BH</sub> = 90 Hz, BH <sub>4</sub> )		

**Selected IR data:** Nujol2417 ν(B-H<sub>t</sub>), 2402 ν(B-H<sub>t</sub>),1705 ν(B-H<sub>b</sub>), 1695 ν(B-H<sub>b</sub>)

6.2.12 Characterising Data for  $[V\{(\eta-C_5H_4)C_2Me_4(\eta-C_5H_4)\}(\eta^2-BH_4)]$  (12)**Appearance:**

Violet solid

 $C_{16}H_{24}BV = 278.12$ 

X-ray crystal structure: Appendix E

<b>Elemental Analyses:</b>	C %	H %
Required:	69.1	8.7
Found:	68.9	8.5

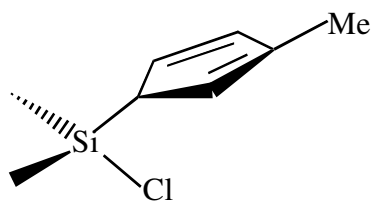
<b><math>^1H</math> NMR:</b>	$C_6D_5CD_3$ 500 MHz
	6.47 (m., 4H, $C_5H_4$ )
	4.74 (m., 4H, $C_5H_4$ )
	0.25 (m., 12H, $C_2Me_4$ )
	-9.10 (br. d., 4H, $BH_4$ )

<b><math>^{13}C\{^1H\}</math> NMR:</b>	$C_6D_5CD_3$ 125 MHz
	108.11 (s., $C_5H_4$ )
	88.12 (s., $C_5H_4$ )
	42.89 (s., $C_2Me_4$ )
	26.08 (s., $C_2Me_4$ )

<b><math>^{11}B</math> NMR:</b>	$C_6D_5CD_3$ 160 MHz
	26.5 (qn., $^1J_{BH} = 86$ Hz, $BH_4$ )

**Selected IR data:** Nujol

2450  $\nu(B-H_t)$ , 2410  $\nu(B-H_t)$ ,  
 1750  $\nu(B-H_b)$ , 1627  $\nu(B-H_b)$

6.2.13 Characterising Data for [(C<sub>5</sub>H<sub>4</sub>Me)SiMe<sub>2</sub>Cl] (13)**Appearance:**

Yellow oil

C<sub>8</sub>H<sub>13</sub>ClSi = 172.73

**<sup>1</sup>H NMR:** CD<sub>2</sub>Cl<sub>2</sub> 500 MHz

6.56 (s., 1H, C<sub>5</sub>H<sub>4</sub>)

6.49 (s., 1H, C<sub>5</sub>H<sub>4</sub>)

6.12 (s., 1H, C<sub>5</sub>H<sub>4</sub>)

3.55 (s., 1H, C<sub>5</sub>H<sub>4</sub>)

2.06 (s., 3H, C<sub>5</sub>H<sub>4</sub>Me)

0.27 (s., 3H, SiMe<sub>2</sub>)

0.22 (s., 3H, SiMe<sub>2</sub>)

**<sup>13</sup>C{<sup>1</sup>H} NMR:** CD<sub>2</sub>Cl<sub>2</sub> 125 MHz

146.20 (s, C<sub>5</sub>H<sub>4</sub>)

138.83 (s, C<sub>5</sub>H<sub>4</sub>)

135.64 (s, C<sub>5</sub>H<sub>4</sub>)

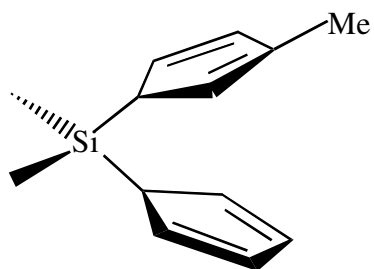
129.21 (s, C<sub>5</sub>H<sub>4</sub>)

55.09 (s, C<sub>ipso</sub> C<sub>5</sub>H<sub>4</sub>)

17.77 (s, C<sub>5</sub>H<sub>4</sub>Me)

3.12 (s, SiMe<sub>2</sub>)

2.60 (s, SiMe<sub>2</sub>)

**6.2.14 Characterising Data for [(C<sub>5</sub>H<sub>4</sub>Me)SiMe<sub>2</sub>(C<sub>5</sub>H<sub>5</sub>)] (14)****Appearance:**

Orange oil

 $C_{13}H_{18}Si = 202.37$ 

**<sup>1</sup>H NMR:** CDCl<sub>3</sub> 500 MHz

δ 6.63 – 6.60 (m., 7H, C<sub>5</sub>H<sub>4</sub> and C<sub>5</sub>H<sub>5</sub>)

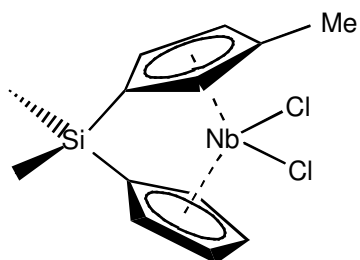
δ 2.96 (s., 1H, C<sub>5</sub>H<sub>4</sub> or C<sub>5</sub>H<sub>5</sub>)

δ 2.85 (s., 1H, C<sub>5</sub>H<sub>4</sub> or C<sub>5</sub>H<sub>5</sub>)

δ 2.06 (s., 3H, C<sub>5</sub>H<sub>4</sub>Me)

δ -0.29 (s., 3H, SiMe<sub>2</sub>)

δ -0.31 (s., 3H, SiMe<sub>2</sub>)

**6.2.15 Characterising Data for [Nb{(η-C<sub>5</sub>H<sub>3</sub>Me)SiMe<sub>2</sub>(η-C<sub>5</sub>H<sub>4</sub>)Cl<sub>2</sub>] (15)****Appearance:**

Brown solid

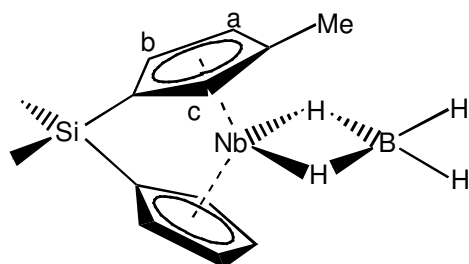
$$C_{13}H_{16}Cl_2NbSi = 364.17$$

<b>Elemental Analyses:</b>	C %	H %
Required:	42.9	4.4
Found:	38.3	4.4

<b>Mass Spectrometry:</b>	m/z = 328	(M-Cl) <sup>+</sup>	45 %
	m/z = 293	(M-2Cl) <sup>+</sup>	30 %

**ESR:** CH<sub>2</sub>Cl<sub>2</sub>, 10 lines,  $g_{iso} = 2.007$ ,  $1a^{93}Nb|_{iso} = 109$  G

### 6.2.16 Characterising Data for $[\text{Nb}\{(\eta\text{-C}_5\text{H}_3\text{Me})\text{SiMe}_2(\eta\text{-C}_5\text{H}_4)\}(\eta^2\text{-BH}_4)]$ (16)



**Appearance:**

Green solid

$\text{C}_{13}\text{H}_{20}\text{BNbSi} = 308.10$

Elemental Analyses: C %		H %
Required:	50.7	6.5
Found:	50.1	6.9

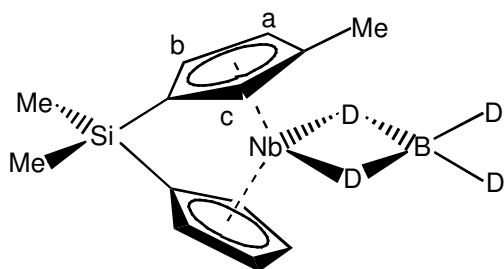
$^1\text{H}$ NMR:	$\text{C}_6\text{D}_5\text{CD}_3$ 500 MHz	$^{13}\text{C}\{^1\text{H}\}$ NMR: $\text{C}_6\text{D}_5\text{CD}_3$ 125 MHz
	6.04 (s., 1H, $\text{C}_5\text{H}_4$ )	113.18 (s., $\text{C}_5\text{H}_3$ , $\text{C}_a$ )
	5.89 (s., 1H, $\text{C}_5\text{H}_4$ )	112.60 (s., $\text{C}_5\text{H}_4$ )
	5.74 (s., 1H, $\text{C}_5\text{H}_3$ , $\text{H}_a$ )	110.72 (s., $\text{C}_5\text{H}_4$ )
	5.36 (s., 1H, $\text{C}_5\text{H}_3$ , $\text{H}_b$ )	87.27 (s., $\text{C}_5\text{H}_3$ , $\text{C}_c$ )
	5.27 (s., 1H, $\text{C}_5\text{H}_4$ )	87.10 (s., $\text{C}_5\text{H}_4$ )
	5.17 (s., 1H, $\text{C}_5\text{H}_4$ )	86.37 (s., $\text{C}_5\text{H}_4$ )
	4.95 (s., 1H, $\text{C}_5\text{H}_3$ , $\text{H}_c$ )	85.52 (s., $\text{C}_5\text{H}_3$ , $\text{C}_b$ )
	1.95 (s., 3H, $\text{C}_5\text{H}_3\text{Me}$ )	73.91 (s., $\text{C}_{ipso}$ )
	-0.72 (s., 3H, $\text{SiMe}_2$ )	73.33 (s., $\text{C}_{ipso}$ )
	-0.79 (s., 3H, $\text{SiMe}_2$ )	20.10 (s., $\text{C}_5\text{H}_3\text{Me}$ )
		-6.58 (s., 3H, $\text{SiMe}_2$ )
$^{11}\text{B}$ NMR:	$\text{C}_6\text{D}_5\text{CD}_3$ 160 MHz	-7.11 (s., 3H, $\text{SiMe}_2$ )
	22.0 (q. $^1J_{\text{BH}}$ 88 Hz, $\text{BH}_4$ )	

**Selected IR data:** Nujol

2468  $\nu(\text{B-H}_t)$ , 2423  $\nu(\text{B-H}_t)$ ,

1700  $\nu(\text{B-H}_b)$ , 1649  $\nu(\text{B-H}_b)$

### 6.2.17 Characterising Data for $[\text{Nb}\{(\eta\text{-C}_5\text{H}_3\text{Me})\text{SiMe}_2(\eta\text{-C}_5\text{H}_4)\}(\eta^2\text{-BD}_4)]$ (17)



**Appearance:**

Green solid

$\text{C}_{13}\text{H}_{16}\text{BD}_4\text{NbSi} = 312.13$

Elemental Analyses: C %	H/D %
Required: 50.7	7.8
Found: 50.0	7.7

**$^1\text{H}$  NMR:**  $\text{C}_6\text{D}_5\text{CD}_3$  500 MHz

5.95 (s., 1H, $\text{C}_5\text{H}_4$ )
5.80 (s., 1H, $\text{C}_5\text{H}_4$ )
5.64 (s., 1H, $\text{C}_5\text{H}_3$ , $\text{H}_a$ )
5.27 (s., 1H, $\text{C}_5\text{H}_3$ , $\text{H}_b$ )
5.26 (s., 1H, $\text{C}_5\text{H}_4$ )
5.18 (s., 1H, $\text{C}_5\text{H}_4$ )
4.80 (s., 1H, $\text{C}_5\text{H}_3$ , $\text{H}_c$ )
1.86 (s., 3H, $\text{C}_5\text{H}_3\text{Me}$ )
-0.72 (s., 3H, $\text{SiMe}_2$ )
-0.78 (s., 3H, $\text{SiMe}_2$ )

**$^{13}\text{C}\{^1\text{H}\}$  NMR:**  $\text{C}_6\text{D}_5\text{CD}_3$  125 MHz

113.24 (s., $\text{C}_5\text{H}_3$ , $\text{C}_a$ )
112.97 (s., $\text{C}_5\text{H}_4$ )
110.80 (s., $\text{C}_5\text{H}_4$ )
87.12 (s., $\text{C}_5\text{H}_3$ , $\text{C}_c$ )
86.57 (s., $\text{C}_5\text{H}_4$ )
86.37 (s., $\text{C}_5\text{H}_4$ )
85.53 (s., $\text{C}_5\text{H}_3$ , $\text{C}_b$ )
73.84 (s., $\text{C}_{ipso}$ )
73.27 (s., $\text{C}_{ipso}$ )
20.09 (s., $\text{C}_5\text{H}_3\text{Me}$ )
-6.58 (s., 3H, $\text{SiMe}_2$ )
-7.12 (s., 3H, $\text{SiMe}_2$ )

**$^2\text{H}$  NMR:**  $\text{C}_6\text{H}_5\text{CH}_3$  76.75 MHz

-4.82 (br. s, $\text{BD}_4$ )
-------------------------------

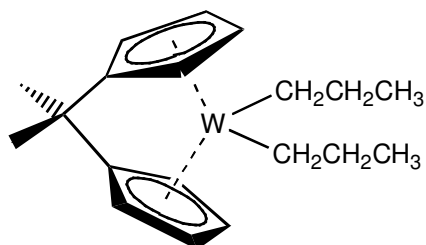
**$^{11}\text{B}$  NMR:**  $\text{C}_6\text{D}_5\text{CD}_3$  160 MHz

21.9 (br. s, $\text{BD}_4$ )
------------------------------

**Selected IR data:** Nujol

1861  $\nu(\text{B-D}_t)$ , 1779  $\nu(\text{B-D}_t)$ ,  
1255  $\nu(\text{B-D}_b)$

## 6.3 Characterising Data for Chapter 3

6.3.1 Characterising Data for  $[W\{(\eta-C_5H_4)CMe_2(\eta-C_5H_4)\}\{(CH_2)_2CH_3\}_2]$   
(18)**Appearance:**

Yellow solid

 $C_{19}H_{28}W = 440.28$ 

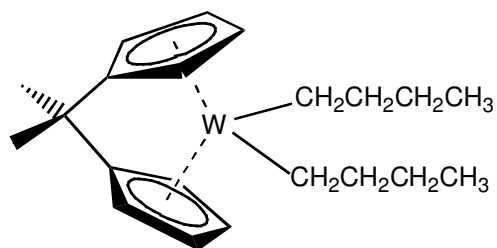
<b>Elemental Analysis:</b>	C %	H %
Required:	51.8	6.4
Found:	50.9	6.5

<b>Mass Spectrometry:</b>	m/z = 440	(M) <sup>+</sup>	45 %
	m/z = 355	(M-2{(CH <sub>2</sub> ) <sub>2</sub> CH <sub>3</sub> }) <sup>+</sup>	100 %

<b><sup>1</sup>H NMR:</b>	C <sub>6</sub> D <sub>6</sub> 500 MHz
	4.28 (m., 4H, C <sub>5</sub> H <sub>4</sub> )
	4.19 (m., 4H, C <sub>5</sub> H <sub>4</sub> )
	1.45 (m., 4H, CH <sub>2</sub> CH <sub>2</sub> CH <sub>3</sub> )
	1.21 (t., 6H, CH <sub>2</sub> CH <sub>2</sub> CH <sub>3</sub> )
	0.55 (m., 4H, CH <sub>2</sub> CH <sub>2</sub> CH <sub>3</sub> )
	0.43 (m., 6H, CMe <sub>2</sub> )

<b><sup>13</sup>C{<sup>1</sup>H} NMR:</b>	C <sub>6</sub> D <sub>6</sub> 125 MHz
	104.26 (s., C <sub>5</sub> H <sub>4</sub> )
	67.19 (s., C <sub>5</sub> H <sub>4</sub> )
	51.43 (s., C <sub>ipso</sub> C <sub>5</sub> H <sub>4</sub> )
	31.13 (s., CMe <sub>2</sub> )
	22.32 (s., CMe <sub>2</sub> )
	22.19 (s., (CH <sub>2</sub> ) <sub>2</sub> CH <sub>3</sub> )
	5.83 (s., (CH <sub>2</sub> ) <sub>2</sub> CH <sub>3</sub> )
	1.04 (s., (CH <sub>2</sub> ) <sub>2</sub> CH <sub>3</sub> )

### 6.3.2 Characterising Data for $[W\{(\eta-C_5H_4)CMe_2(\eta-C_5H_4)\}\{(CH_2)_3CH_3\}_2]$ (19)

**Appearance:**

Yellow solid

 $C_{21}H_{32}W = 468.33$ 

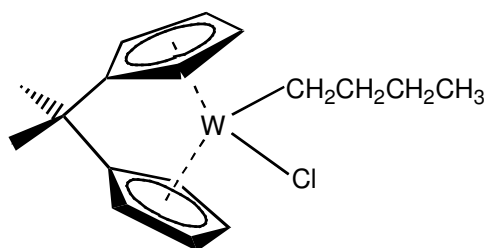
Elemental Analysis:	C %	H %
Required:	53.9	6.9
Found:	53.6	6.7

Mass Spectrometry:	m/z = 468	(M) <sup>+</sup>	86 %
	m/z = 411	(M - {(CH <sub>2</sub> ) <sub>3</sub> CH <sub>3</sub> }) <sup>+</sup>	20 %
	m/z = 355	(M - 2{(CH <sub>2</sub> ) <sub>3</sub> CH <sub>3</sub> }) <sup>+</sup>	100 %

<sup>1</sup> H NMR:	C <sub>6</sub> D <sub>6</sub> 500 MHz
	4.31 (m., 4H, C <sub>5</sub> H <sub>4</sub> )
	4.22 (m., 4H, C <sub>5</sub> H <sub>4</sub> )
	1.52 (m., 8H, CH <sub>2</sub> (CH <sub>2</sub> ) <sub>2</sub> CH <sub>3</sub> )
	1.16 (t., 6H, CH <sub>2</sub> (CH <sub>2</sub> ) <sub>2</sub> CH <sub>3</sub> )
	0.56 (m., 4H, CH <sub>2</sub> (CH <sub>2</sub> ) <sub>2</sub> CH <sub>3</sub> )
	0.42 (s., 6H, CMe <sub>2</sub> )

<sup>13</sup> C{ <sup>1</sup> H} NMR:	C <sub>6</sub> D <sub>6</sub> 125 MHz
	104.29 (s., C <sub>5</sub> H <sub>4</sub> )
	67.20 (s., C <sub>5</sub> H <sub>4</sub> )
	51.70 (s., C <sub>ipso</sub> C <sub>5</sub> H <sub>4</sub> )
	40.72 (s., (CH <sub>2</sub> ) <sub>3</sub> CH <sub>3</sub> )
	30.38 (s., CMe <sub>2</sub> )
	30.35 (s., (CH <sub>2</sub> ) <sub>3</sub> CH <sub>3</sub> )
	22.20 (s., CMe <sub>2</sub> )
	5.92 (s., (CH <sub>2</sub> ) <sub>3</sub> CH <sub>3</sub> )
	1.73 (s., (CH <sub>2</sub> ) <sub>3</sub> CH <sub>3</sub> )

### 6.3.3 Characterising Data for $[W\{(\eta-C_5H_4)CMe_2(\eta-C_5H_4)\}\{(CH_2)_3CH_3\}Cl]$ (20)

**Appearance:**

Pale red solid

$$C_{17}H_{23}ClW = 446.67$$

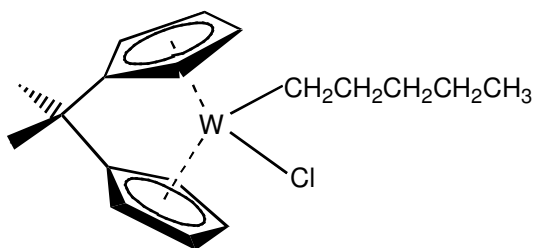
<b>Elemental Analysis:</b>	C %	H %
Required:	45.7	5.2
Found:	46.2	5.5

<b>Mass Spectrometry:</b>	m/z = 446	(M) <sup>+</sup>	55 %
	m/z = 411	(M-Cl) <sup>+</sup>	25 %
	m/z = 389	(M-((CH <sub>2</sub> ) <sub>3</sub> CH <sub>3</sub> )) <sup>+</sup>	100 %

<b><sup>1</sup>H NMR:</b>	C <sub>6</sub> D <sub>6</sub> 500 MHz
	5.20 (m., 2H, C <sub>5</sub> H <sub>4</sub> )
	4.71 (m., 2H, C <sub>5</sub> H <sub>4</sub> )
	4.68 (m., 2H, C <sub>5</sub> H <sub>4</sub> )
	3.96 (m., 2H, C <sub>5</sub> H <sub>4</sub> )
	1.55 (m., 2H, CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> CH <sub>3</sub> )
	1.37 (m., 2H, CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> CH <sub>3</sub> )
	1.31 (t., 2H, CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> CH <sub>3</sub> )
	1.15 (t., 3H, CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> CH <sub>3</sub> )
	0.35 (s., 3H, CMe <sub>2</sub> )
	0.03 (s., 3H, CMe <sub>2</sub> )

<b><sup>13</sup>C{<sup>1</sup>H} NMR:</b>	C <sub>6</sub> D <sub>6</sub> 125 MHz
	115.76 (s., C <sub>5</sub> H <sub>4</sub> )
	106.44 (s., C <sub>5</sub> H <sub>4</sub> )
	70.15 (s., C <sub>5</sub> H <sub>4</sub> )
	69.59 (s., C <sub>5</sub> H <sub>4</sub> )
	57.92 (s., C <sub>ipso</sub> C <sub>5</sub> H <sub>4</sub> )
	39.69 (s., (CH <sub>2</sub> ) <sub>3</sub> CH <sub>3</sub> )
	29.59 (s., (CH <sub>2</sub> ) <sub>3</sub> CH <sub>3</sub> )
	22.39 (s., (CH <sub>2</sub> ) <sub>2</sub> CH <sub>3</sub> )
	21.76 (s., CMe <sub>2</sub> )
	21.61 (s., CMe <sub>2</sub> )
	14.12 (s., (CH <sub>2</sub> ) <sub>3</sub> CH <sub>3</sub> )

### 6.3.4 Characterising Data for $[W\{(\eta-C_5H_4)CMe_2(\eta-C_5H_4)\}\{(CH_2)_4CH_3\}Cl]$ (21)

**Appearance:**

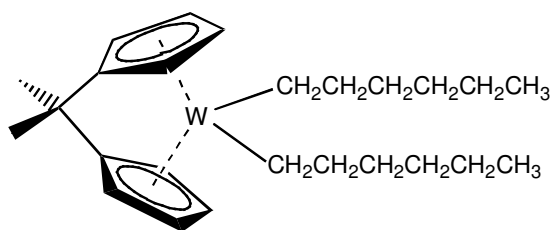
Pale red solid

 $C_{18}H_{25}ClW = 460.70$ 

<b>Elemental Analysis:</b>		C %	H %	Cl %
Required:		46.9	5.5	7.7
Found:		46.3	5.8	7.2
<b>Mass Spectrometry:</b>		$m/z = 462$	$(M)^+$	18 %
		$m/z = 389$	$(M - \{(CH_2)_4CH_3\})^+$	100 %
<b><math>^1H</math> NMR:</b>	$C_6D_6$ 500 MHz			
	5.19 (m., 2H, $C_5H_4$ )			115.57 (s., $C_5H_4$ )
	4.70 (m., 4H, $C_5H_4$ )			106.17 (s., $C_5H_4$ )
	3.97 (m., 2H, $C_5H_4$ )			70.18 (s., $C_5H_4$ )
	1.55 (m., 2H, $CH_2CH_2$			69.48 (s., $C_5H_4$ )
	$CH_2CH_3$ )			57.93 (s., $C_{ipso} C_5H_4$ )
	1.53 (m., 2H, $CH_2CH_2$			39.03 (s., $(CH_2)_4CH_3$ )
	$CH_2CH_2CH_3$ )			36.88 (s., $(CH_2)_4CH_3$ )
	1.42 (t., 2H, $CH_2CH_2$			29.42 (s., $CMe_2$ )
	$CH_2CH_3$ )			22.72 (s., $(CH_2)_4CH_3$ )
	1.29 (m., 2H, $CH_2CH_2$			21.78 (s., $CMe_2$ )
	$CH_2CH_2CH_3$ )			21.62 (s., $CMe_2$ )
	1.11 (t., 3H, $CH_2CH_2$			14.48 (s., $(CH_2)_4CH_3$ )
	$CH_2CH_3$ )			1.38 (s., $(CH_2)_4CH_3$ )
	0.35 (s., 3H, $CMe_2$ )			
	0.05 (s., 3H, $CMe_2$ )			

<b><math>^{13}C\{^1H\}</math> NMR:</b>		$C_6D_6$ 125 MHz
		115.57 (s., $C_5H_4$ )
		106.17 (s., $C_5H_4$ )
		70.18 (s., $C_5H_4$ )
		69.48 (s., $C_5H_4$ )
		57.93 (s., $C_{ipso} C_5H_4$ )
		39.03 (s., $(CH_2)_4CH_3$ )
		36.88 (s., $(CH_2)_4CH_3$ )
		29.42 (s., $CMe_2$ )
		22.72 (s., $(CH_2)_4CH_3$ )
		21.78 (s., $CMe_2$ )
		21.62 (s., $CMe_2$ )
		14.48 (s., $(CH_2)_4CH_3$ )
		1.38 (s., $(CH_2)_4CH_3$ )

### 6.3.5 Characterising Data for $[W\{(\eta-C_5H_4)CMe_2(\eta-C_5H_4)\}\{(CH_2)_5CH_3\}_2]$ (22)

**Appearance:**

Yellow solid

 $C_{25}H_{40}W = 524.44$ 

<b>Elemental Analysis:</b>	C %	H %
Required:	57.3	7.7
Found:	56.5	7.5

<b>Mass Spectrometry:</b>	m/z = 524	(M) <sup>+</sup>	85 %
	m/z = 439	(M - {(CH <sub>2</sub> ) <sub>5</sub> CH <sub>3</sub> }) <sup>+</sup>	17 %
	m/z = 355	(M - 2{(CH <sub>2</sub> ) <sub>5</sub> CH <sub>3</sub> }) <sup>+</sup>	100 %

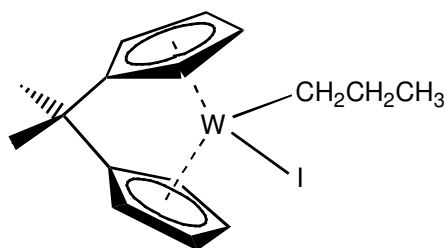
**<sup>1</sup>H NMR:** C<sub>6</sub>D<sub>6</sub> 500 MHz

4.35 (m., 4H, C <sub>5</sub> H <sub>4</sub> )
4.25 (m., 4H, C <sub>5</sub> H <sub>4</sub> )
1.52 (m., 16H, CH <sub>2</sub> (CH <sub>2</sub> ) <sub>4</sub> CH <sub>3</sub> )
1.06 (t., 6H, CH <sub>2</sub> (CH <sub>2</sub> ) <sub>4</sub> CH <sub>3</sub> )
0.58 (m., 4H, CH <sub>2</sub> (CH <sub>2</sub> ) <sub>2</sub> CH <sub>3</sub> )
0.44 (s., 6H, CMe <sub>2</sub> )

**<sup>13</sup>C{<sup>1</sup>H} NMR:** C<sub>6</sub>D<sub>6</sub> 125 MHz

104.25 (s., C <sub>5</sub> H <sub>4</sub> )
67.23 (s., C <sub>5</sub> H <sub>4</sub> )
51.48 (s., C <sub>ipso</sub> C <sub>5</sub> H <sub>4</sub> )
38.08 (s., (CH <sub>2</sub> ) <sub>5</sub> CH <sub>3</sub> )
37.29 (s., (CH <sub>2</sub> ) <sub>5</sub> CH <sub>3</sub> )
32.07 (s., (CH <sub>2</sub> ) <sub>5</sub> CH <sub>3</sub> )
30.78 (s., CMe <sub>2</sub> )
23.28 (s., (CH <sub>2</sub> ) <sub>5</sub> CH <sub>3</sub> )
22.19 (s., CMe <sub>2</sub> )
14.28 (s., (CH <sub>2</sub> ) <sub>5</sub> CH <sub>3</sub> )
2.15 (s., (CH <sub>2</sub> ) <sub>5</sub> CH <sub>3</sub> )

6.3.6 Characterising Data for  $[W\{(\eta-C_5H_4)CMe_2(\eta-C_5H_4)\}\{(CH_2)_2CH_3\}I]$   
(23)

**Appearance:**

Pale red solid

$$C_{16}H_{21}IW = 524.10$$

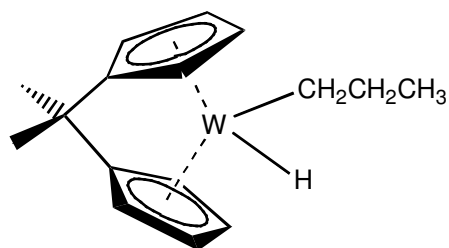
Elemental Analysis:	C %	H %
Required:	36.7	4.1
Found:	36.8	4.3

Mass Spectrometry:	m/z = 524	(M) <sup>+</sup>	62%
	m/z = 481	(M - {(CH <sub>2</sub> ) <sub>2</sub> CH <sub>3</sub> }) <sup>+</sup>	85%

<sup>1</sup> H NMR:	C <sub>6</sub> D <sub>6</sub> 500 MHz
	5.57 (m., 2H, C <sub>5</sub> H <sub>4</sub> )
	4.39 (m., 2H, C <sub>5</sub> H <sub>4</sub> )
	4.37 (m., 2H, C <sub>5</sub> H <sub>4</sub> )
	3.83 (m., 2H, C <sub>5</sub> H <sub>4</sub> )
	1.53 (m., 2H, CH <sub>2</sub> CH <sub>2</sub> CH <sub>3</sub> )
	1.18 (m., 2H, CH <sub>2</sub> CH <sub>2</sub> CH <sub>3</sub> )
	1.15 (t., 3H, CH <sub>2</sub> CH <sub>2</sub> CH <sub>3</sub> )
	0.22 (s., 3H, CMe <sub>2</sub> )
	0.09 (s., 3H, CMe <sub>2</sub> )

<sup>13</sup> C{ <sup>1</sup> H} NMR:	C <sub>6</sub> D <sub>6</sub> 125 MHz
	111.55 (s., C <sub>5</sub> H <sub>4</sub> )
	105.58 (s., C <sub>5</sub> H <sub>4</sub> )
	70.62 (s., C <sub>5</sub> H <sub>4</sub> )
	68.38 (s., C <sub>5</sub> H <sub>4</sub> )
	57.32 (s., C <sub>ipso</sub> C <sub>5</sub> H <sub>4</sub> )
	32.62 (s., (CH <sub>2</sub> ) <sub>2</sub> CH <sub>3</sub> )
	22.39 (s., (CH <sub>2</sub> ) <sub>2</sub> CH <sub>3</sub> )
	22.21 (s., CMe <sub>2</sub> )
	21.83 (s., CMe <sub>2</sub> )
	-0.53 (s., (CH <sub>2</sub> ) <sub>2</sub> CH <sub>3</sub> )

**6.3.7 Characterising Data for  $[W\{(\eta-C_5H_4)CMe_2(\eta-C_5H_4)\}\{(CH_2)_2CH_3\}H]$   
(24)**

**Appearance:**

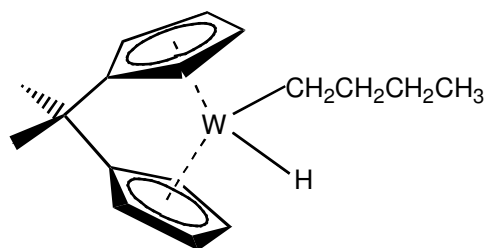
Yellow solid

 $C_{16}H_{22}W = 398.20$ 

Elemental Analysis:	C %	H %
Required:	48.3	5.6
Found:	47.8	6.1

$^1H$ NMR:	$C_6D_6$ 500 MHz	$^{13}C\{^1H\}$ NMR:	$C_6D_6$ 125 MHz
	5.10 (m., 2H, $C_5H_4$ )		103.79 (s., $C_5H_4$ )
	4.48 (m., 2H, $C_5H_4$ )		86.89 (s., $C_5H_4$ )
	4.29 (m., 2H, $C_5H_4$ )		70.21 (s., $C_5H_4$ )
	3.78 (m., 2H, $C_5H_4$ )		62.82 (s., $C_5H_4$ )
	1.36 (m., 2H, $CH_2CH_2$ $CH_3$ )		57.32 (s., $C_{ipso} C_5H_4$ )
	1.13 (t., 3H, $CH_2 CH_2$ $CH_3$ )		33.30 (s., $(CH_2)_2CH_3$ )
	1.04 (m., 2H, $CH_2 CH_2$ $CH_3$ )		22.35 (s., $(CH_2)_2CH_3$ )
	0.56 (s., 3H, $CMe_2$ )		22.02 (s., $CMe_2$ )
	0.29 (s., 3H, $CMe_2$ )		21.96 (s., $CMe_2$ )
	-6.47 (m., $^1J_{WH} = 50$ Hz, 1H, $WH_2$ )		-14.99 (s., $(CH_2)_2CH_3$ )

6.3.8 Characterising Data for  $[W\{(\eta-C_5H_4)CMe_2(\eta-C_5H_4)\}\{(CH_2)_3CH_3\}H]$   
(25)

**Appearance:**

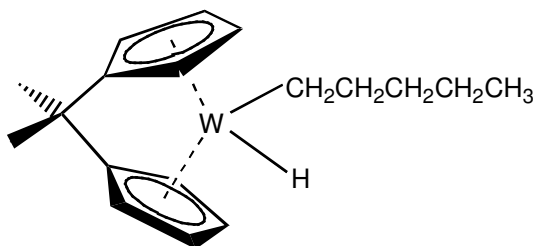
Yellow solid

 $C_{17}H_{24}W = 412.23$ 

Elemental Analysis: C %			H %
Required:	49.5		5.8
Found:	49.5		5.9

$^1H$ NMR:	$C_6D_6$ 500 MHz	$^{13}C\{^1H\}$ NMR: $C_6D_6$ 125 MHz
	5.10 (m., 2H, $C_5H_4$ )	103.94 (s., $C_5H_4$ )
	4.48 (m., 2H, $C_5H_4$ )	86.81 (s., $C_5H_4$ )
	4.29 (m., 2H, $C_5H_4$ )	70.25 (s., $C_5H_4$ )
	3.78 (m., 2H, $C_5H_4$ )	62.77 (s., $C_5H_4$ )
	1.45 (m., 2H, $CH_2CH_2$ $CH_2CH_3$ )	53.88 (s., $C_{ipso} C_5H_4$ )
	1.36 (m., 2H, $CH_2CH_2$ $CH_2CH_3$ )	42.75 (s., $(CH_2)_3CH_3$ )
	1.11 (t., 3H, $CH_2CH_2$ $CH_2CH_3$ )	30.50 (s., $(CH_2)_2CH_3$ )
	1.02 (m., 2H, $CH_2CH_2$ $CH_2CH_3$ )	22.03 (s., $CMe_2$ )
	0.55 (s., 3H, $CMe_2$ )	21.95 (s., $CMe_2$ )
	0.29 (s., 3H, $CMe_2$ )	14.08 (s., $(CH_2)_3CH_3$ )
	-6.46 (m., $^1J_{WH} = 50$ Hz, 1H, $WH_2$ )	-12.15 (s., $(CH_2)_3CH_3$ )

### 6.3.9 Characterising Data for $[W\{(\eta-C_5H_4)CMe_2(\eta-C_5H_4)\}\{(CH_2)_4CH_3\}H]$ (26)

**Appearance:**

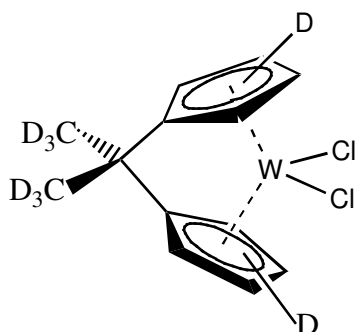
Yellow solid

 $C_{18}H_{26}W = 426.25$ 

<b>Elemental Analysis:</b>	C %	H %
Required:	50.7	6.2
Found:	49.7	5.9

<b>Mass Spectrometry:</b>	m/z = 426	(M-H) <sup>+</sup>	25%
	m/z = 355	(M-((CH <sub>2</sub> ) <sub>4</sub> CH <sub>3</sub> )-H) <sup>+</sup>	100%

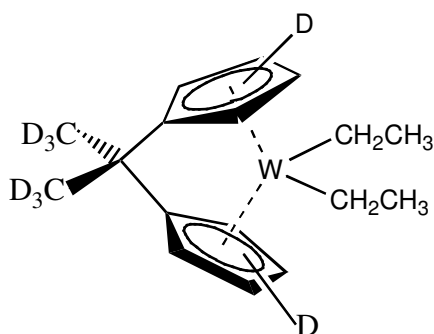
<b><sup>1</sup>H NMR:</b>	$C_6D_6$ 500 MHz	<b><sup>13</sup>C{<sup>1</sup>H} NMR:</b>	$C_6D_6$ 125 MHz
	5.12 (m., 2H, C <sub>5</sub> H <sub>4</sub> )		103.78 (s., C <sub>5</sub> H <sub>4</sub> )
	4.51 (m., 2H, C <sub>5</sub> H <sub>4</sub> )		86.84 (s., C <sub>5</sub> H <sub>4</sub> )
	4.32 (m., 2H, C <sub>5</sub> H <sub>4</sub> )		70.20 (s., C <sub>5</sub> H <sub>4</sub> )
	3.80 (m., 2H, C <sub>5</sub> H <sub>4</sub> )		62.74 (s., C <sub>5</sub> H <sub>4</sub> )
	1.49 (m., 2H, CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> CH <sub>3</sub> )		53.88 (s., C <sub>ipso</sub> C <sub>5</sub> H <sub>4</sub> )
	1.38 (m., 2H, CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> CH <sub>3</sub> )		40.10 (s., (CH <sub>2</sub> ) <sub>4</sub> CH <sub>3</sub> )
	1.36 (m., 2H, CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> CH <sub>3</sub> )		40.04 (s., (CH <sub>2</sub> ) <sub>4</sub> CH <sub>3</sub> )
	1.07 (t., 3H, CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> CH <sub>3</sub> )		31.70 (s., CMe <sub>2</sub> )
	1.01 (m., 2H, CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> CH <sub>2</sub> CH <sub>3</sub> )		22.78 (s., (CH <sub>2</sub> ) <sub>4</sub> CH <sub>3</sub> )
	0.46 (s., 3H, CMe <sub>2</sub> )		22.05 (s., CMe <sub>2</sub> )
	0.20 (s., 3H, CMe <sub>2</sub> )		21.96 (s., CMe <sub>2</sub> )
	-6.49 (m., <sup>1</sup> J <sub>WH</sub> = 48 Hz, 1H, WH)		14.51 (s., (CH <sub>2</sub> ) <sub>4</sub> CH <sub>3</sub> )
			-11.83 (s., (CH <sub>2</sub> ) <sub>4</sub> CH <sub>3</sub> )

6.3.10 Characterising Data for  $[W\{(\eta-C_5D_4)C(CD_3)_2(\eta-C_5D_4)\}Cl_2]$  (27)**Appearance:**

Purple solid

 $C_{12}Cl_2D_{14}W = 427.08$ 

**$^2H$  NMR:**  $C_6H_6$  76.75 MHz  
 5.96 (br. s., 4D,  $C_5D_4$ )  
 5.44 (br. s., 4D,  $C_5D_4$ )  
 0.70 (s., 6D,  $C(CD_3)_2$ )

6.3.11 Characterising Data for  $[W\{(\eta-C_5D_4)C(CD_3)_2(\eta-C_5D_4)\}\{(CH_2CH_3)_2\}]$  (28)**Appearance:**

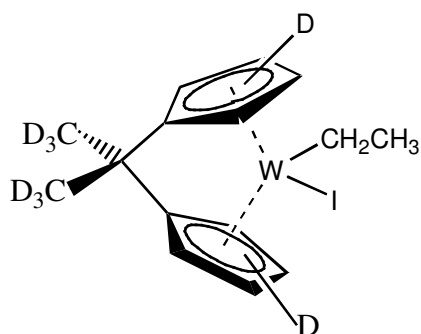
Yellow solid

 $C_{17}H_{10}D_{14}W = 426.31$ 

**$^1H$  NMR:**  $C_6D_6$  500 MHz  
 1.59 (t., 6H,  $CH_2CH_3$ )  
 0.58 (q., 4H,  $CH_2CH_3$ )

**$^2H$  NMR:**  $C_6H_6$  76.75 MHz  
 4.30 (s., 4D,  $C_5D_4$ )  
 4.26 (s., 4D,  $C_5D_4$ )  
 0.33 (s., 6D,  $C(CD_3)_2$ )

**6.3.12 Characterising Data for  $[W\{(\eta-C_5D_4)C(CD_3)_2(\eta-C_5D_4)\}\{CH_2CH_3\}I]$   
(29)**

**Appearance:**

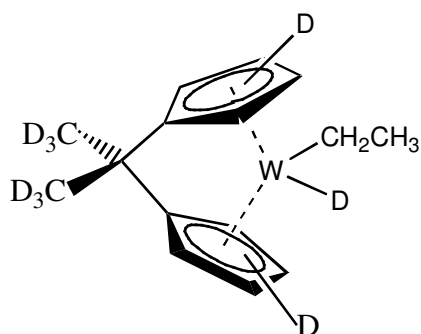
Pale red solid

 $C_{15}H_5D_{14}IW = 524.16$ 

**$^1H$  NMR:**  $C_6D_6$  500 MHz  
 1.59 (q., 2H,  $\underline{CH}_2CH_3$ )  
 1.14 (t., 3H,  $CH_2\underline{CH}_3$ )

**$^2H$  NMR:**  $C_6H_6$  76.75 MHz  
 5.54 (m., 2D,  $C_5D_4$ )  
 4.39 (m., 2D,  $C_5D_4$ )  
 4.37 (m., 2D,  $C_5D_4$ )  
 3.67 (m., 2D,  $C_5D_4$ )  
 0.20 (s., 3D,  $C(CD_3)_2$ )  
 0.07 (s., 3D,  $C(CD_3)_2$ )

**6.3.13 Characterising Data for  $[W\{(\eta-C_5D_4)C(CD_3)_2(\eta-C_5D_4)\}\{CH_2CH_3\}D]$   
(30)**

**Appearance:**

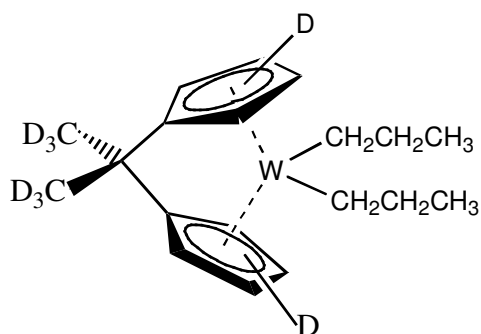
Yellow solid

 $C_{15}H_5D_{15}W = 399.26$ 

**$^1H$  NMR:**  $C_6D_6$  500 MHz  
 1.58 (t., 3H,  $CH_2CH_3$ )  
 1.12 (q., 2H,  $CH_2CH_3$ )

**$^2H$  NMR:**  $C_6H_6$  76.75 MHz  
 5.08 (s., 2D,  $C_5D_4$ )  
 4.48 (s., 2D,  $C_5D_4$ )  
 4.28 (s., 2D,  $C_5D_4$ )  
 3.75 (s., 2D,  $C_5D_4$ )  
 0.46 (s., 3D,  $C(CD_3)_2$ )  
 0.19 (s., 3D,  $C(CD_3)_2$ )  
 -6.44 (s., 1D, WD)

**6.3.14 Characterising Data for  $[\text{W}\{(\eta\text{-C}_5\text{D}_4)\text{C}(\text{CD}_3)_2(\eta\text{-C}_5\text{D}_4)\}\{(\text{CH}_2)_2\text{CH}_3\}_2]$   
(18a)**

**Appearance:**

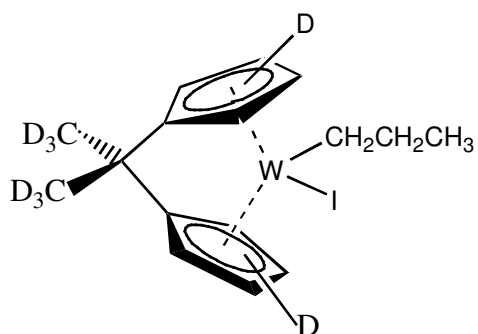
Yellow solid

 $\text{C}_{19}\text{H}_{14}\text{D}_{14}\text{W} = 454.37$ 

**$^1\text{H}$  NMR:**  $\text{C}_6\text{D}_6$  500 MHz  
 1.46 (m., 4H,  $\text{CH}_2\text{CH}_2$   
 $\text{CH}_3$ )  
 1.21 (t., 6H,  $\text{CH}_2\text{CH}_2$   
 $\text{CH}_3$ )  
 0.54 (m., 4H,  $\text{CH}_2\text{CH}_2$   
 $\text{CH}_3$ )

**$^2\text{H}$  NMR:**  $\text{C}_6\text{H}_6$  76.75 MHz  
 4.24 (br. s., 8D,  $\text{C}_5\text{D}_4$ )  
 0.42 (s., 6D,  $\text{C}(\text{CD}_3)_2$ )

**6.3.15 Characterising Data for  $[W\{(\eta-C_5D_4)C(CD_3)_2(\eta-C_5D_4)\}\{(CH_2)_2CH_3\}I]$   
(23a)**

**Appearance:**

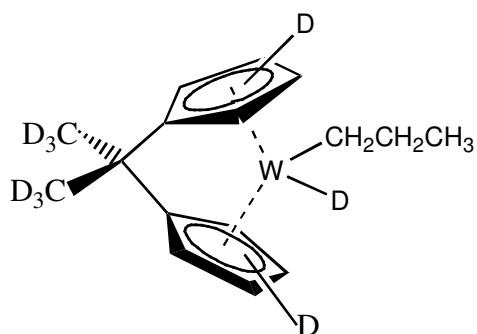
Pale red solid

 $C_{16}H_7D_{14}IW = 538.18$ 

**$^1H$  NMR:**  $C_6D_6$  500 MHz  
 1.52 (m., 2H,  $CH_2CH_2CH_3$ )  
 1.17 (m., 2H,  $CH_2CH_2CH_3$ )  
 1.14 (t., 3H,  $CH_2CH_2CH_3$ )

**$^2H$  NMR:**  $C_6H_6$  76.75 MHz  
 5.55 (m., 2D,  $C_5D_4$ )  
 4.39 (m., 4D,  $C_5D_4$ )  
 3.82 (m., 2D,  $C_5D_4$ )  
 0.18 (s., 3D,  $C(CD_3)_2$ )  
 0.07 (s., 3D,  $C(CD_3)_2$ )

**6.3.16 Characterising Data for  $[W\{(\eta-C_5D_4)C(CD_3)_2(\eta-C_5D_4)\}\{(CH_2)_2CH_3\}D]$   
(24a)**

**Appearance:**

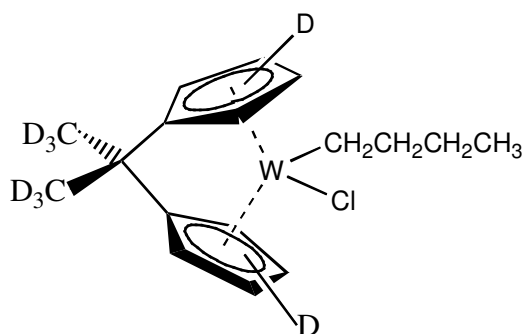
Yellow solid

 $C_{16}H_7D_{15}W = 413.29$ 

**$^1H$  NMR:**  $C_6D_6$  500 MHz  
 1.37 (m., 2H,  $CH_2CH_2CH_3$ )  
 1.16 (t., 3H,  $CH_2CH_2CH_3$ )  
 1.04 (m., 2H,  $CH_2CH_2CH_3$ )

**$^2H$  NMR:**  $C_6H_6$  76.75 MHz  
 5.09 (s., 2D,  $C_5D_4$ )  
 4.51 (s., 2D,  $C_5D_4$ )  
 4.29 (s., 2D,  $C_5D_4$ )  
 3.77 (s., 2D,  $C_5D_4$ )  
 0.52 (s., 3D,  $C(CD_3)_2$ )  
 0.26 (s., 3D,  $C(CD_3)_2$ )  
 -6.44 (s., 1D, WD)

**6.3.17 Characterising Data for  $[W\{(\eta-C_5D_4)C(CD_3)_2(\eta-C_5D_4)\}\{(CH_2)_3CH_3\}Cl]$  (20a)**



**Appearance:**

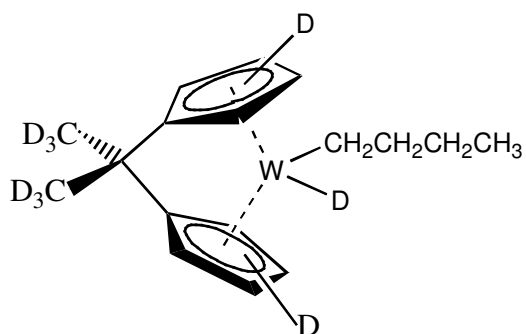
Pale red solid

$C_{17}H_{19}ClD_{14}W = 460.76$

**$^1H$  NMR:**  $C_6D_6$  500 MHz  
 1.55 (m., 2H,  $\underline{CH_2}CH_2$   
 $\underline{CH_2}CH_3$ )  
 1.37 (m., 2H,  $CH_2\underline{CH_2}$   
 $CH_2CH_3$ )  
 1.28 (t., 3H,  $\underline{CH_2}CH_2$   
 $CH_2CH_3$ )  
 1.15 (t., 3H,  $CH_2CH_2$   
 $CH_2\underline{CH_3}$ )

**$^2H$  NMR:**  $C_6H_6$  75 MHz  
 5.20 (s., 2D,  $C_5D_4$ )  
 4.73 (s., 2D,  $C_5D_4$ )  
 4.68 (s., 2D,  $C_5D_4$ )  
 3.98 (s., 2D,  $C_5D_4$ )  
 0.38 (s., 3D,  $C(CD_3)_2$ )  
 0.06 (s., 3D,  $C(CD_3)_2$ )

**6.3.18 Characterising Data for  $[W\{(\eta-C_5D_4)C(CD_3)_2(\eta-C_5D_4)\}\{(CH_2)_3CH_3\}D]$   
(25a)**

**Appearance:**

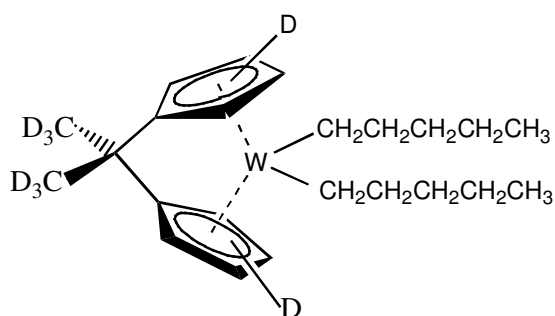
Yellow solid

 $C_{17}H_7D_{15}W = 427.32$ 

**$^1H$  NMR:**  $C_6D_6$  500 MHz  
 1.43 (m., 2H,  $CH_2CH_2$   
 $CH_2CH_3$ )  
 1.35 (m., 2H,  $CH_2CH_2$   
 $CH_2CH_3$ )  
 1.16 (t., 3H,  $CH_2CH_2$   
 $CH_2CH_3$ )  
 1.01 (m., 2H,  $CH_2CH_2$   
 $CH_2CH_3$ )

**$^2H$  NMR:**  $C_6H_6$  76.75 MHz  
 5.05 (s., 2D,  $C_5D_4$ )  
 4.47 (s., 2D,  $C_5D_4$ )  
 4.26 (s., 2D,  $C_5D_4$ )  
 3.77 (s., 2D,  $C_5D_4$ )  
 0.56 (s., 3D,  $C(CD_3)_2$ )  
 0.27 (s., 3D,  $C(CD_3)_2$ )  
 -6.45 (s., 1D, WD)

**6.3.19 Characterising Data for  $[W\{(\eta-C_5D_4)C(CD_3)_2(\eta-C_5D_4)\}\{(CH_2)_4CH_3\}_2]$   
(21a)**

**Appearance:**

Yellow solid

 $C_{23}H_{22}D_{14}W = 510.47$ 

**$^1H$  NMR:**  $C_6D_6$  500 MHz

1.59 (m., 4H,  $CH_2CH_2$   
 $CH_2CH_2CH_3$ )

1.50 (m., 4H,  $CH_2CH_2$   
 $CH_2CH_2CH_3$ )

1.42 (m., 4H,  $CH_2CH_2$   
 $CH_2CH_2CH_3$ )

1.09 (t., 6H,  $CH_2CH_2$   
 $CH_2CH_2CH_3$ )

0.75 (m., 4H,  $CH_2CH_2$   
 $CH_2CH_2CH_3$ )

**$^2H$  NMR:**  $C_6H_6$  76.75 MHz

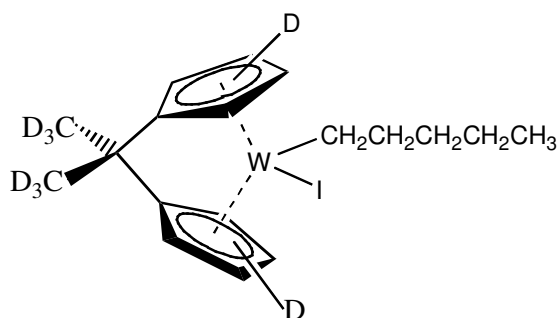
4.31 (s., 4D,  $C_5D_4$ )

4.27 (s., 4D,  $C_5D_4$ )

0.38 (s., 3D,  $C(CD_3)_2$ )

0.19 (s., 3D,  $C(CD_3)_2$ )

**6.3.20 Characterising Data for  $[W\{(\eta-C_5D_4)C(CD_3)_2(\eta-C_5D_4)\}\{(CH_2)_4CH_3\}I]$  (21b)**

**Appearance:**

Pale red solid

 $C_{18}H_{11}D_{14}IW = 566.24$ 

**$^1H$  NMR:**  $C_6D_6$  500 MHz

1.56 (m., 2H,  $CH_2CH_2$   
 $CH_2CH_2CH_3$ )

1.53 (m., 2H,  $CH_2CH_2$   
 $CH_2CH_2CH_3$ )

1.40 (t., 2H,  $CH_2CH_2$   
 $CH_2CH_3$ )

1.29 (m., 2H,  $CH_2CH_2$   
 $CH_2CH_2CH_3$ )

1.09 (t., 3H,  $CH_2CH_2$   
 $CH_2CH_2CH_3$ )

**$^2H$  NMR:**  $C_6H_6$  76.75 MHz

5.19 (s., 2D,  $C_5D_4$ )

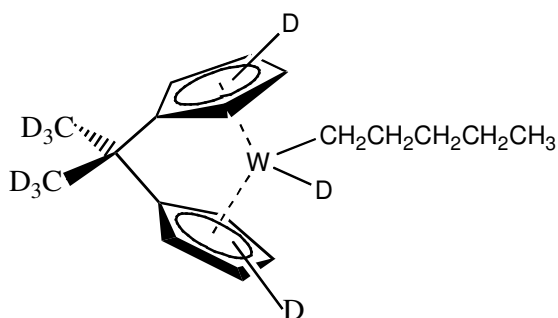
4.70 (s., 4D,  $C_5D_4$ )

3.96 (s., 2D,  $C_5D_4$ )

0.35 (s.,  $C(CD_3)_2$ )

0.05 (s.,  $C(CD_3)_2$ )

**6.3.21 Characterising Data for  $[W\{(\eta-C_5D_4)C(CD_3)_2(\eta-C_5D_4)\}\{(CH_2)_4CH_3\}D]$   
(26a)**

**Appearance:**

Yellow solid

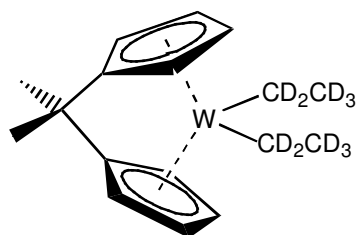
 $C_{18}H_{11}D_{15}W = 441.34$ 

**$^1H$  NMR:**  $C_6D_6$  500 MHz  
 1.54 (m., 2H,  $CH_2CH_2$   
 $CH_2CH_2CH_3$ )  
 1.42 (m., 2H,  $CH_2CH_2$   
 $CH_2CH_2CH_3$ )  
 1.30 (m., 2H,  $CH_2CH_2$   
 $CH_2CH_2CH_3$ )  
 1.10 (t., 3H,  $CH_2CH_2$   
 $CH_2CH_2CH_3$ )  
 1.05 (m., 2H,  $CH_2CH_2$   
 $CH_2CH_2CH_3$ )

**$^2H$  NMR:**  $C_6H_6$  76.75 MHz  
 5.11 (m., 2D,  $C_5D_4$ )  
 4.46 (m., 2D,  $C_5D_4$ )  
 4.32 (m., 2D,  $C_5D_4$ )  
 3.80 (m., 2D,  $C_5D_4$ )  
 0.47 (s., 3D,  $C(CD_3)_2$ )  
 0.24 (s., 3D,  $C(CD_3)_2$ )  
 -6.52 (s. 1D, WD)

6.3.22 Characterising Data for  $[W\{(\eta-C_5H_4)CMe_2(\eta-C_5H_4)\}\{CD_2CD_3\}_2]$ 

(31)

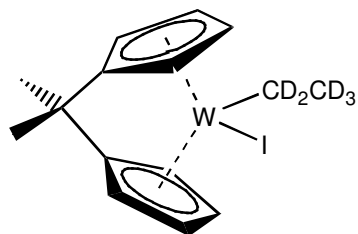
**Appearance:**

Yellow solid

 $C_{17}H_{14}D_{10}W = 422.29$ 

**$^1H$  NMR:**  $C_6D_5CD_3$  500 MHz  
 4.27 (m., 4H,  $C_5H_4$ )  
 4.19 (m., 4H,  $C_5H_4$ )  
 0.44 (s., 6H,  $CMe_2$ )

**$^2H$  NMR:**  $C_6H_6$  76.75 MHz  
 1.60 (s., 6D,  $CD_2CD_3$ )  
 0.62 (s., 4D,  $CD_2CD_3$ )

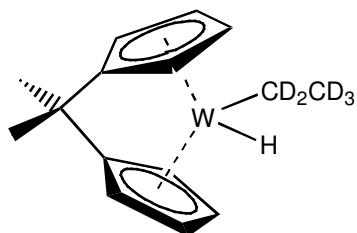
6.3.23 Characterising Data for  $[W\{(\eta-C_5H_4)CMe_2(\eta-C_5H_4)\}\{CD_2CD_3\}I]$  (32)**Appearance:**

Pale red solid

 $C_{15}H_{14}D_5IW = 515.10$ 

**$^1H$  NMR:**  $C_6D_6$  500 MHz  
 5.56 (s., 2H,  $C_5H_4$ )  
 4.38 (s., 4H,  $C_5H_4$ )  
 3.84 (s., 2H,  $C_5H_4$ )  
 0.17 (s., 3H,  $CMe_2$ )  
 0.04 (s., 3H,  $CMe_2$ )

**$^2H$  NMR:**  $C_6H_6$  76.75 MHz  
 1.57 (s., 2D,  $CD_2CD_3$ )  
 1.34 (s., 3D,  $CD_2CD_3$ )

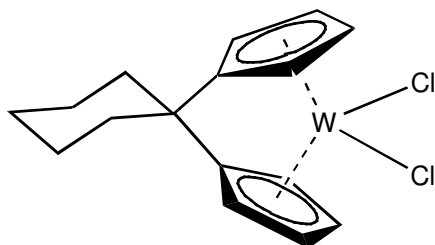
6.3.24 Characterising Data for  $[W\{(\eta-C_5H_4)CMe_2(\eta-C_5H_4)\}\{CD_2CD_3\}H]$  (33)**Appearance:**

Yellow solid

$$C_{15}H_{15}D_5W = 389.20$$

<b><math>^1H</math> NMR:</b>	$C_6D_6$ 500 MHz	<b><math>^2H</math> NMR:</b>	$C_6H_6$ 76.75 MHz
	5.11 (s., 2H, $C_5H_4$ )		1.58 (s., 3D, $CD_2CD_3$ )
	4.48 (s., 2H, $C_5H_4$ )		1.13 (s., 2D, $CD_2CD_3$ )
	4.29 (s., 2H, $C_5H_4$ )		
	3.77 (s., 2H, $C_5H_4$ )		
	0.53 (s., 3H, $CMe_2$ )		
	0.29 (s., 3H, $CMe_2$ )		
	-6.47 (s. 1H, $^1J_{WH} = 31$ Hz, WH)		

## 6.4 Characterising Data for Chapter 4

6.4.1 Characterising Data for  $[W\{(\eta-C_5H_4)C(C_5H_{10})(\eta-C_5H_4)\}Cl_2]$  (34)**Appearance:**

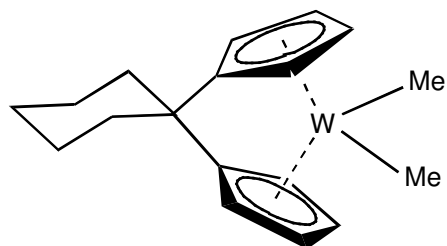
Dark brown solid

$$C_{16}H_{18}Cl_2W = 465.07$$

<b>Elemental Analysis:</b>	C %	H %
Required:	40.6	4.2
Found:	41.3	3.9

<b>Mass Spectrometry:</b>	m/z = 465	(M) <sup>+</sup>	45 %
	m/z = 429	(M-Cl) <sup>+</sup>	92 %

<b><sup>1</sup>H NMR:</b>	CD <sub>2</sub> Cl <sub>2</sub> 500 MHz	<b><sup>13</sup>C{<sup>1</sup>H} NMR:</b>	CD <sub>2</sub> Cl <sub>2</sub> 125 MHz
	5.95 (m., 4H, C <sub>5</sub> H <sub>4</sub> )		119.67 (s, C <sub>5</sub> H <sub>4</sub> )
	5.45 (m., 4H, C <sub>5</sub> H <sub>4</sub> )		72.46 (s, C <sub>5</sub> H <sub>4</sub> )
	1.46 (m., 6H, C(C <sub>5</sub> H <sub>10</sub> ))		68.93 (s, C <sub>ipso</sub> C <sub>5</sub> H <sub>4</sub> )
	1.12 (m., 4H, C(C <sub>5</sub> H <sub>10</sub> ))		34.67 (s, C(C <sub>5</sub> H <sub>10</sub> ))
			31.24 (s., C(C <sub>5</sub> H <sub>10</sub> ))
			25.68 (s, C(C <sub>5</sub> H <sub>10</sub> ))
			20.88 (s, C(C <sub>5</sub> H <sub>10</sub> ))

6.4.2 Characterising Data for  $[W\{(\eta-C_5H_4)C(C_5H_{10})(\eta-C_5H_4)\}Me_2]$  (35)**Appearance:**

Orange solid

 $C_{18}H_{24}W = 424.24$ 

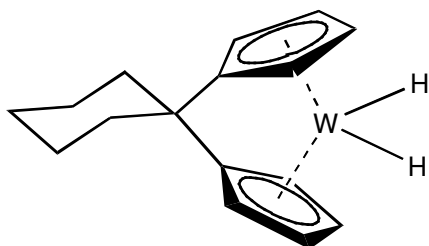
X-ray crystal structure: Appendix F

<b>Elemental Analysis:</b>	C %	H %
Required:	51.0	5.7
Found:	51.2	5.7

<b>Mass Spectrometry:</b>	m/z = 424	(M) <sup>+</sup>	100 %
	m/z = 409	(M-Me) <sup>+</sup>	51 %
	m/z = 395	(M-2Me) <sup>+</sup>	16%

<b><sup>1</sup>H NMR:</b>	C <sub>6</sub> D <sub>5</sub> CD <sub>3</sub> 500 MHz
	4.41 (m., 4H, C <sub>5</sub> H <sub>4</sub> )
	4.06 (m., 4H, C <sub>5</sub> H <sub>4</sub> )
	1.57 (m., 6H, C(C <sub>5</sub> H <sub>10</sub> ))
	0.94 (m., 4H, C(C <sub>5</sub> H <sub>10</sub> ))
	0.27 (m., 6H, WMe <sub>2</sub> )

<b><sup>13</sup>C{<sup>1</sup>H} NMR:</b>	C <sub>6</sub> D <sub>5</sub> CD <sub>3</sub> 125 MHz
	105.68 (s., C <sub>5</sub> H <sub>4</sub> )
	68.58 (s., C <sub>5</sub> H <sub>4</sub> )
	71.46 (s., C <sub>ipso</sub> C <sub>5</sub> H <sub>4</sub> )
	52.43 (s., C(C <sub>5</sub> H <sub>10</sub> ))
	37.35 (s., C(C <sub>5</sub> H <sub>10</sub> ))
	32.36 (s., C(C <sub>5</sub> H <sub>10</sub> ))
	-21.71 (s., WMe <sub>2</sub> )

6.4.3 Characterising Data for  $[W\{(\eta-C_5H_4)C(C_5H_{10})(\eta-C_5H_4)\}H_2]$  (36)**Appearance:**

Yellow solid

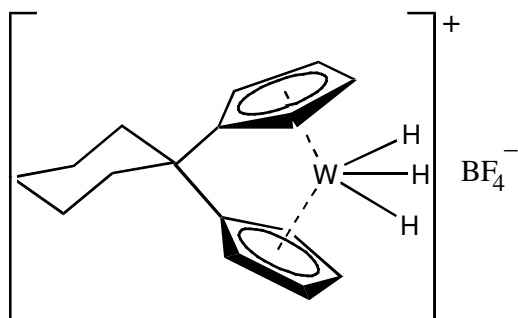
 $C_{16}H_{20}W = 396.18$ 

<b>Elemental Analysis:</b>	C %	H %
Required:	48.5	5.1
Found:	48.8	5.1

**Mass Spectrometry:**  $m/z = 394$   $(M-2H)^+$  100%

**$^1H$  NMR:**  $C_6D_6$  500 MHz  
 4.95 (m., 4H,  $C_5H_4$ )  
 4.13 (m., 4H,  $C_5H_4$ )  
 1.32 (m., 4H,  $C(C_5H_{10})$ )  
 1.22 (m., 2H,  $C(C_5H_{10})$ )  
 0.90 (t., 4H,  $C(C_5H_{10})$ )  
 -7.39 (m.  $^1J_{W-H} = 94$  Hz,  
 2H,  $WH_2$ )

**Selected IR data:** Nujol1828  $\nu(W-H)$ 1816  $\nu(W-H)$

6.4.4 Characterising Data for  $[W\{(\eta-C_5H_4)C(C_5H_{10})(\eta-C_5H_4)\}H_3][BF_4]$  (37)**Appearance:**

White solid

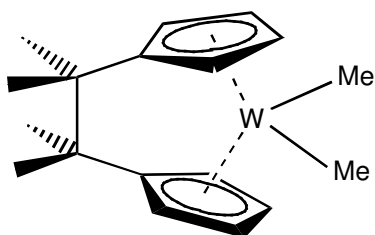
 $C_{16}H_{21}BF_4W = 484.00$ 

**$^1H$  NMR:**  $CD_2Cl_2$  500 MHz  
 5.76 (m., 4H,  $C_5H_4$ )  
 5.61 (m., 4H,  $C_5H_4$ )  
 1.91 (m., 4H,  $C(C_5H_{10})$ )  
 1.77 (m., 2H,  $C(C_5H_{10})$ )  
 1.67 (t., 4H,  $C(C_5H_{10})$ )  
 -3.10 (m., 3H,  $WH_3$ )

**$^{13}C\{^1H\}$  NMR:**  $CD_2Cl_2$  125 MHz  
 93.81 (s.,  $C_5H_4$ )  
 82.32 (s.,  $C_5H_4$ )  
 71.46 (s.,  $C_{ipso} C_5H_4$ )  
 53.88 (s.,  $\underline{C}(C_5H_{10})$ )  
 30.24 (s.,  $C(\underline{C}_5H_{10})$ )  
 24.86 (s.,  $C(\underline{C}_5H_{10})$ )  
 20.15 (s.,  $C(\underline{C}_5H_{10})$ )

**$^{11}B$  NMR:**  $CD_2Cl_2$  160 MHz  
 -6.29 (m.,  $BF_4$ )

**Selected IR data:** Nujol  
 1898  $\nu(W-H)$

6.4.5 Characterising Data for  $[W\{(\eta-C_5H_4)C_2Me_4(\eta-C_5H_4)\}Me_2]$  (38)**Appearance:**

Orange solid

 $C_{18}H_{26}W = 426.25$ 

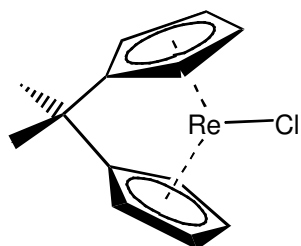
X-ray crystal structure: Appendix G

<b>Elemental Analysis:</b>	C %	H %
Required:	50.7	6.2
Found:	51.2	5.7

<b>Mass Spectrometry:</b>	m/z = 426	(M) <sup>+</sup>	100 %
	m/z = 411	(M-Me) <sup>+</sup>	55 %
	m/z = 395	(M-2Me) <sup>+</sup>	26 %

<b><sup>1</sup>H NMR:</b>	C <sub>6</sub> D <sub>6</sub> 500 MHz
	4.96 (m., 4H, C <sub>5</sub> H <sub>4</sub> )
	4.17 (m., 4H, C <sub>5</sub> H <sub>4</sub> )
	0.66 (s., 12H, C <sub>2</sub> Me <sub>4</sub> )
	0.29 (m., 6H, WMe <sub>2</sub> )

<b><sup>13</sup>C{<sup>1</sup>H} NMR:</b>	C <sub>6</sub> D <sub>6</sub> 125 MHz
	105.68 (s., C <sub>ipso</sub> C <sub>5</sub> H <sub>4</sub> )
	104.41 (s., C <sub>5</sub> H <sub>4</sub> )
	72.09 (s., C <sub>5</sub> H <sub>4</sub> )
	47.10 (s., C <sub>2</sub> Me <sub>4</sub> )
	27.64 (s., C <sub>2</sub> Me <sub>4</sub> )
	-26.05 (s., WMe <sub>2</sub> )

6.4.6 Characterising Data for  $[\text{Re}\{(\eta\text{-C}_5\text{H}_4)\text{CMe}_2(\eta\text{-C}_5\text{H}_4)\}\text{Cl}]$  (39)**Appearance:**

Red/orange solid

 $\text{C}_{13}\text{H}_{14}\text{ClRe} = 391.91$ 

X-ray crystal structure: Appendix H

<b>Elemental Analysis:</b>	C %	H %
Required:	39.8	3.6
Found:	39.8	3.9

<b>Mass Spectrometry:</b>	m/z = 392	(M) <sup>+</sup>	85%
	m/z = 357	(M-Cl) <sup>+</sup>	17%

**<sup>1</sup>H NMR:** CD<sub>2</sub>Cl<sub>2</sub> 500 MHz  
 5.29 (m., 4H, C<sub>5</sub>H<sub>4</sub>)  
 4.36 (m., 4H, C<sub>5</sub>H<sub>4</sub>)  
 0.99 (s., 6H, CMe<sub>2</sub>)

**<sup>13</sup>C{<sup>1</sup>H} NMR:** CD<sub>2</sub>Cl<sub>2</sub> 125 MHz  
 83.84 (s, C<sub>5</sub>H<sub>4</sub>)  
 76.18 (s, C<sub>5</sub>H<sub>4</sub>)  
 34.39 (s, C<sub>ipso</sub> or CMe<sub>2</sub>)  
 31.56 (s, C<sub>ipso</sub> or CMe<sub>2</sub>)  
 22.09 (s, CMe<sub>2</sub>)