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Novel carbohydrate-substituted cyclopentadienyls of titanium, molybdenum, manganese and iron

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Abstract

The synthesis of the carbohydrate-substituted cyclopentadiene **1** is obtained by reaction of 3-(*O*-*tert*-butyldimethylsilyl)-1,2-*O*-isopropylidene-5-*O*-(*p*-tolylsulfonyl)-α-D-xylofuranose and cyclopentadienyl sodium in dimethylformamide. Thallation of **1** and reaction with $FeCl_2$ or $CpTiCl_3$ affords the symmetric ferrocene Cp^s_2Fe (**3**) and the mixed titanocene $Cp^sCpTiCl_2$ (**4**), respectively. The manganese monocyclopentadienyl $Cp^sMn(CO)_3$ (**5**) is isolated by reaction of the thallium salt of **1** with $MnBr(CO)_5$. Direct reaction of 1 with the molybdenum tricarbonyl $Mo(CO)_3(NCMe)_3$ followed by treatment with iodoform led to $Cp^sMo(CO)_3I$ (**6**). All new compounds were characterised by elemental analysis, IR and NMR spectroscopy.

Keywords: Sugar; Cyclopentadienyls; Molybdenum; Iron; Titanium; Manganese; Ferrocene

1. Introduction

During the last decade, the introduction of ligands at the cyclopentadienyl (Cp) fragment has become an useful tool to modify the chemical and physical properties of Cp derivatives [1]. Introduction of a carbohydrate into the Cp fragment is an attractive source of optically active ligands, which can open an entrance into the synthesis of new chiral catalysts.

So far, enantiomerically pure complexes derived from carbohydrates have been reported and used in catalysis [2–10] but there are few examples of organometallic complexes containing a carbohydrate-substituted Cp ligand [11–15]. In this paper we decided to explore systems containing a sugar unit in the Cp fragment. We report the preparation of a carbohydrate-substituted cyclopentadiene and its transformation into the corresponding thallium, iron, titanium, manganese and molybdenum complexes.

Besides, the growing importance of organometallic compounds in bio-medical applications could also take advantage of the presence of sugar moieties attached to the Cp ligands that modulate solubility and interaction properties of these molecules with cells.

2. Results and discussion

The carbohydrate-derived cyclopentadiene compound Cp^sH (**1**) is prepared by reaction of 3-(*O*-*tert*-butyldimethylsilyl)-1,2-*O*-isopropylidene-5-*O*-(*p*-tolylsulfonyl)-α-D-xylofuranose [16] and excess of CpNa in DMF at room temperature (see Scheme 1). A yellow oil is obtained after purification by column chromatography. When THF instead of DMF is used as a solvent, the starting materials are recovered unchanged. Similar results are reported in the literature [12]. The ^{1}H-NMR spectrum of **1** gives no enough structural information. A mixture of isomers is present due to the tautomerism of the double bond. Further characterisation of **1** was not possible. Compound **1** is air stable but decomposes slowly at room temperature.

Reaction of the isomeric mixture with one equivalent of thallium ethoxide in hexane at low temperature affords the corresponding thallium salt Cp^sTl (**2**), which is isolated as an insoluble white solid and characterised by elemental analyses (see Scheme 2).

Treatment of the thallium salt with $FeCl_2$ or $CpTiCl_3$ in tetrahydrofuran leads to the symmetric ferrocene Cp^s_2Fe (**3**) isolated as a yellow oil and the mixed titanocene $Cp^sCpTiCl_2$ (**4**) isolated as a red crystalline solid, respectively (see Scheme 2). The ^{1}H-NMR spectrum of **3** shows broad signals at room temperature

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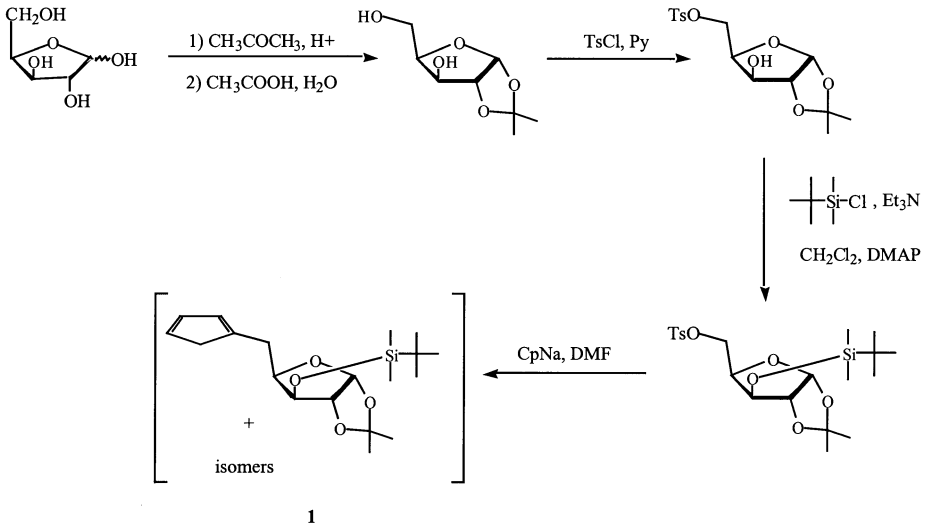


Scheme 1. Synthesis of the carbohydrate-derived cyclopentadiene **1**.

probably due to a fluxional behaviour. When the NMR spectrum is recorded at −80 °C the signals appear with better resolution but not enough to get structural information. Its ^{13}C-NMR spectrum allows the identification of the sugar moiety by the presence of three singlets at δ 106.8, 84.2 and 78.9 ppm, a singlet at δ 87.8 ppm for the C1–C4 (see Fig. 1) and a singlet at δ 31.0 ppm (CH_2, C5) (see Table 2). The ^{1}H-NMR spectrum of 4 shows three multiplets for the Cp ring containing the sugar moiety at δ 6.61–6.57, 6.24–6.21 (two protons) and 5.77–5.70 ppm and one singlet at δ 6.11 ppm for the five protons of the unsubstitued Cp. The sugar moiety gives three doublets at δ 6.08, 4.50 and 78.9 ppm for the H-1, H-2 and H-3, two multiplets at δ 4.68–4.64 and 3.38–3.34 ppm for the H-4, H-5a and H-5b (see Fig. 1), two singlets at δ 1.53 and 1.21 ppm for the isopropylidene group, one singlet at δ 1.01 ppm for the *tert*-butyl protons and two singlets at δ 0.14 and 0.10 ppm for the methyl groups of the *tert*-butyldimethylsilyl group (see Table 1).

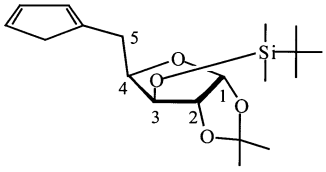
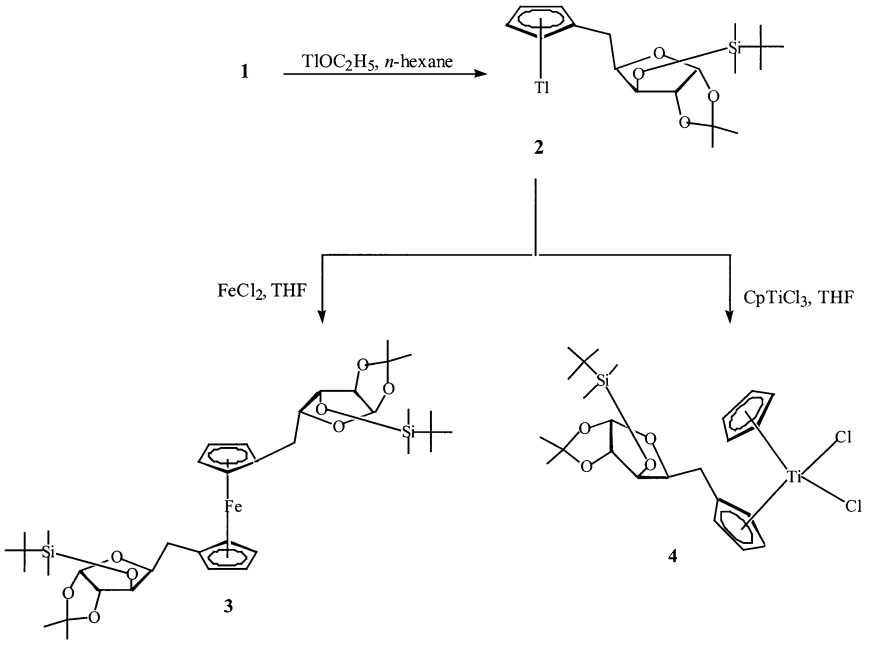


Fig. 1. Labelled carbon atoms of the sugar moiety.



Scheme 2. Synthesis of the carbohydrate-derived Cps **2**, **3** and **4**.

Table 1
^{1}H-NMR chemical shifts for compounds **3**, **4**, **5** and **6**

Compound	^{1}H						
	Cp^s	H5	H1	H2, H3, H4	CH_3-isop	Bu^t	$SiMe_3$
3	4.45–4.25 (m, 4H)	2.55–2.37 (m, 2H)	5.79 (br. s, 1H)	4.01–3.92 (m, 3H)	1.34 (s, 3H) 1.19 (s, 3H)	0.86 (s, 9H)	0.07 (s, 6H)
4	6.61–6.57 (m, 1H) 6.24–6.21 (m, 2H) 5.77–5.70 (m, 1H)	3.38–3.34 (m, 2H)	6.08 (d, 1H)	4.68–4.64 (m, 1H) 4.50 (d, 1H) 4.26 (d, 1H)	1.53 (s, 3H) 1.21 (s, 3H)	1.01 (s, 9H)	0.14 (s, 3H) 0.10 (s, 3H)
5	4.81–4.79 (m, 1H) 4.74–4.72 (m, 2H) 4.66–4.64 (m, 1H)	2.62 (dd, 1H) 2.36–2.29 (m, 1H)	5.85 (d, 1H)	4.37 (d, 1H) 4.14–4.10 (m, 1H) 4.08 (d, 1H)	1.43 (s, 3H) 1.27 (s, 3H)	0.91 (s, 9H)	0.13 (s, 3H) 0.11 (s, 3H)
6	5.63–5.61 (m, 1H) 5.55–5.53 (m, 1H) 5.32–5.31 (m, 2H)	2.84 (dd, 1H) 2.53 (dd, 1H)	5.84 (d, 1H)	4.31 (d, 1H) 4.13–4.09 (m, 1H) 4.04 (d, 1H)	1.41 (s, 3H) 1.24 (s, 3H)	0.85 (s, 9H)	0.08 (s, 3H) 0.07 (s 3H)

Treatment of the thallium salt **1** with one equivalent of $MnBr(CO)_5$ in tetrahydrofuran affords the manganese complex $Cp^sMn(CO)_3$ (**5**) (see Scheme 3). Compound **5** is isolated as a yellow oil very soluble in hydrocarbon solvents. The CO ligands are identified in the IR spectrum by the presence of two strong absorptions at 2019 and 1928 cm^{-1} and in the ^{13}C-NMR spectrum by a resonance at δ 226.4 ppm. Its ^{1}H-NMR spectrum shows three multiplets at δ 4.81–4.79, 4.74–4.72 (two protons) and 4.66–4.64 ppm for the protons of the Cp ring. The signals assigned to the sugar moiety show the same pattern that the observed in complex **4** (see Tables 1 and 2).

Reaction of the carbohydrate-substituted cyclopentadiene Cp^sH (**1**) with one equivalent of $Mo(CO)_3(NCMe)_3$ in toluene, followed by treatment with a stoichiometric amount of CHI_3 in dichloromethane, yields $Cp^sMo(CO)_3I$ (**6**) which is isolated as red crystalline solid (see Scheme 4). The CO groups are observed in the IR spectrum at 2036, 1963, 1884 cm^{-1} and in ^{13}C-NMR at δ 237.2, 220.8 ppm.

3. Conclusion

We have synthesised a series of transition metal derivatives containing a carbohydrated-substituted Cp ligand. The reaction of CpNa with the sugar moiety gives a novel carbohydrate-substituted cyclopentadiene which is transformed into the thallium, iron, manganese, molybdenum and titanium derivatives.

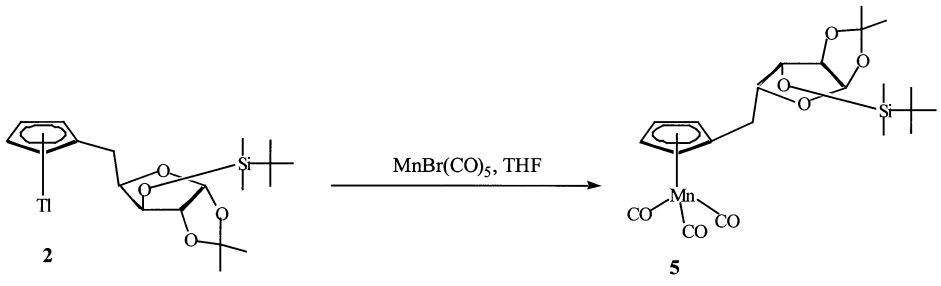
Studies considering the use of these compounds in bio-medical and catalytic applications are currently underway in our laboratory.

4. Experimental

4.1. Materials and procedures

All manipulations are performed in an inert atmosphere using standard Schlenk techniques or in an inert atmosphere glove-box. Solvents are purified by distillation from an appropriate drying/deoxygenation agent. NMR spectra are recorded on a Bruker AMX-300 spectrometer. IR are recorded on a Unicam Mattson model 7000 FTIR spectrometer using KBr pellets. Elemental analyses are performed at ITQB by Maria Conceição Almeida.

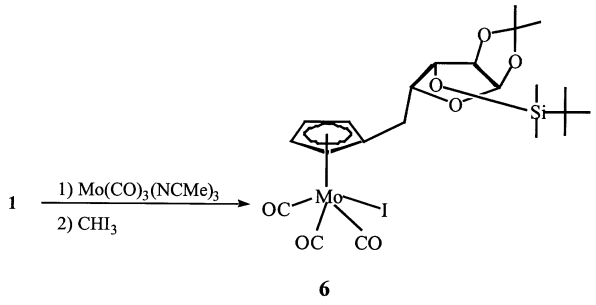
All common chemicals and solvents are purchased from commercial suppliers. The synthesis of $MnBr(CO)_5$ [17], $Mo(CO)_3(NCMe)_3$ [18] and $CpTiCl_3$ [19] was done according to literature procedures.



Scheme 3. Synthesis of the manganese complex **5**.

Table 2
^{13}C-NMR chemical shifts for compounds **3**, **4**, **5** and **6**

Compound	^{13}C						
	Cp^s	C5	C1	C2, C3, C4	CH_3-isop C_q(isop)	Bu^t	$SiMe_3$
3	71.5 C_q not observed	31.0	106.8	87.8 84.2 78.9	28.8 28.2 113.2 (Cq)	27.8 20.2 (Cq)	−2.7
4	135.6 (C_q) 123.9 122.7 116.6 114.6	31.3	105.5	86.4 80.8 77.7	27.1 26.4 111.5 (C_q)	25.9 26.1 (C_q)	−4.9
5	86.6 84.4 C_q not observed	29.2	105.8	83.9 82.4 77.8	27.6 27.1 118.0 (C_q)	26.5 19.0 (C_q)	−4.0
6	111.5 (C_q) 95.7 90.8	29.2	104.8	85.5 81.3 76.7	26.7 26.2 116.5 (C_q)	25.7 18.0 (C_q)	−5.0 −4.7



6

Scheme 4. Synthesis of the molybdenum complex **6**.

4.2. Preparation of Cp^sH (*1*)

A solution of 3-(*O*-*tert*-butyldimethylsilyl)-1,2-*O*-isopropylidene-5-*O*-(*p*-tolylsulfonyl)-α-D-xylofuranose (3.7 g, 8.1 mmol) in DMF (20 ml) is added to CpNa (2.2 g, 25 mmol), previously dissolved in DMF (8 ml), at −30 °C. The reaction mixture is stirred at low temperature for 15 min and is then allowed to warm up to room temperature (r.t.) and stirred for further 2 h. Distillated water (40 ml) is added to the mixture to destroy the excess of CpNa. The mixture is extracted with dichloromethane (2 × 100 ml) and the organic layer is dried with Na_2SO_4. All the volatiles are evaporated under vacuum to yield a brown residue which is purified by column chromatography on silica (AcOEt/*n*-hexane 1:8) to yield a yellow oil. Yield 2.15 g (75%).

4.3. Preparation of Cp^sTl (*2*)

To a precooled solution of Cp^sH (1.2 g, 3.41 mmol) in *n*-hexane (50 ml) is added dropwise thallium ethoxide (0.24 ml, 3.41 mmol) at −30 °C. The reaction mixture is stirred for 1 h at −30 °C and is then allowed to warm up to r.t. and stirred for a further 16 h. A white precipitate is isolated by filtration and washed with Et_2O to yield the title compound **2**. Yield 1.57 g (83%). Anal. Calc. for $C_{19}H_{31}SiO_4Tl$: C, 41.05; H, 5.62. Found: C, 40.81; H, 5.39%.

4.4. Preparation of Cp^s_2Fe (*3*)

A mixture of $FeCl_2$ (0.056 g, 0.44 mmol) and Cp^sTl (0.5 g, 0.89 mmol) is weighed in a Schlenk tube in the drybox. Precooled THF (30 ml) is added to the solids and the reaction mixture is stirred at −30 °C for 1 h. The stirring is prolonged at r.t. for a further 16 h. All the volatiles are evaporated under vacuum and the residue is extracted with hexane to yield **5** as a waxy solid. Yield 0.58 g (85%). Anal. Calc. for $C_{38}H_{62}FeO_8Si_2$: C, 60.14; H, 8.23. Found: C, 60.36; H, 8.55%. ^{1}H-NMR (CD_2Cl_2, 300 MHz, −80 °C, δ ppm): 5.79 (br.s, 1H, H-1); 4.45–4.25 (m, 4H, Cp^s); 4.01–3.92 (m, 3H, H-2, H-3, H-4); 2.55–2.37 (m, 2H, H-5a, H-5b); 1.34 (s, 3H, CH_3-isop); 1.19 (s, 3H, CH_3-isop); 0.86 (s, 9H, *tert*-Bu); 0.07 (s, 6H, CH_3-silyl). ^{13}C-NMR (CD_2Cl_2, 300 MHz, 25 °C, δ ppm): 113.2 (s, Cq, isop); 106.8 (s, CH, C1); 87.8 (s, CH, C2 or C3 or C4); 84.2, 78.9 (s, CH, C2 or C3 or C4); 71.5 (s, CH, Cp); 31.0 (s, CH_2, C5); 28.8, 28.2 (s, CH_3-isop); 27.8 (s, *tert*-Bu); 20.2 (s, Cq, *tert*-Bu); −2.7 (s, CH_3-silyl); (Cq, Cp^s not observed).

4.5. Preparation of $Cp^sCpTiCl_2$ (*4*)

A mixture of $CpTiCl_3$ (0.24 g, 1.08 mmol) and Cp^sTl (0.6 g, 1.08 mmol) is weighed in a Schlenk tube in the glovebox and placed in a cold bath at −30 °C. Precooled THF (30 ml) is added and the reaction mixture is stirred for 30 min at low temperature. The

colour of the solution rapidly changes from yellow to orange and finally a red solution and a white precipitate is formed. The mixture is then allowed to warm up to r.t. and stirred for a further 2 h. All the volatiles are removed under vacuum and the residue is extracted in dichloromethane and washed with cold *n*-hexane to give **6** as a red solid. Yield 0.42 g (72%). Anal. Calc. for $C_{24}H_{36}O_4SiTiCl_2$: C, 53.84; H, 6.78. Found: C, 53.69; H, 6.58%. ^{1}H-NMR ($CDCl_3$, 300 MHz, 25 °C, δ ppm): 6.61–6.57 (m, 1H, Cp^s); 6.24–6.21 (m, 2H, Cp^s); 6.11 (s, 5H, Cp); 6.08 (d, 1H, H-1, $J_{1,2} = 3.9$ Hz); 5.77–5.70 (m, 1H, Cp^s); 4.68–4.64 (m, 1H, H-4, $J_{4,5a} = 8.7$ Hz, $J_{4,5b} = 3.3$ Hz); 4.50 (d, 1H, H-2); 4.26 (d, 1H, H-3, $J_{3,4} = 2.7$ Hz); 3.38–3.34 (m, 2H, H-5a, H-5b, $J_{5a,5b} = 5.1$ Hz); 1.53 (s, 3H, CH_3-isop); 1.21 (s, 3H, CH_3-isop); 1.01 (s, 9H, *tert*-Bu); 0.14 (s, 3H, CH_3-silyl); 0.10 (s, 3H, CH_3-silyl). ^{13}C-NMR (C_6D_6, 300 MHz, 25 °C δ ppm): 135.6 (s, Cq, Cp^s); 123.9, 122.7 (s, CH, Cp^s); 119.3 (s, CH, Cp); 116.6, 114.6 (s, CH, Cp^s); 111.5 (s, Cq, isop); 105.5 (s, CH, C1); 86.4, 80.8, 77.7 (s, CH, C2–C4); 31.3 (s, CH_2, C5); 27.1, 26.4 (s, CH_3-isop); 25.9 (s, *tert*-Bu); 26.1 (s, Cq, *tert*-Bu); −4.9 (s, CH_3-silyl). Selected IR (KBr, cm^{-1}): 3112, 2954, 2922, 1373.

4.6. Preparation of $Cp^sMn(CO)_3$ (5)

Solid $MnBr(CO)_5$ (0.14 g, 0.54 mmol) is added to a stirred solution of Cp^sTl (0.3 g, 0.54 mmol) in THF (40 ml) at r.t. The reaction mixture is stirred for 16 h and the solvent is removed under vacuum. The residue is extracted in dichloromethane to yield the title compound **4** as a waxy yellow solid. Yield 0.19 g (73%). Anal. Calc. for $C_{22}H_{31}MnO_7Si$: C, 53.87; H, 6.37. Found: C, 53.51; H, 6.15%. ^{1}H-NMR (CD_2Cl_2, 300 MHz, 25 °C δ ppm): 5.85 (d, 1H, H-1, $J_{1,2} = 3.6$ Hz); 4.81–4.79 (m, 1H, Cp^s); 4.74–4.72 (m, 2H, Cp^s); 4.66–4.64 (m, 1H, Cp^s); 4.37 (d, 1H, H-2); 4.14–4.10 (m, 1H, H-4); 4.08 (d, 1H, H-3, $J_{3,4} = 2.7$ Hz); 2.62 (dd, 1H, H-5a, $J_{5a,5b} = 15$ Hz, $J_{5a,4} = 9.9$ Hz); 2.36–2.29 (m, 1H, H-5b); 1.43 (s, 3H, CH_3-isop); 1.27 (s, 3H, CH_3-isop); 0.91 (s, 9H, *tert*-Bu); 0.13 (s, 3H, CH_3-silyl); 0.11 (s, 3H, CH_3-silyl). ^{13}C-NMR ($CDCl_3$, 300 MHz, 25 °C δ ppm): 226.4 (s, CO); 118.0 (s, Cq, isop); 105.8 (s, CH, C1); 86.6, 84.4 (s, CH, Cp^s); (Cq, Cp^s not observed, probably overlapped with the signals of the CH of the carbons of the Cp); 83.9, 82.4, 77.8 (s, CH, C2–C4); 29.2 (s, CH_2, C5); 27.6, 27.1 (s, CH_3-isop); 26.5 (s, *tert*-Bu); 19.0 (s, Cq, *tert*-Bu); −4.0 (s, CH_3-silyl). Selected IR (KBr, cm^{-1}): 2019, 1928, vs, ν(CO).

4.7. Preparation of $Cp^sMo(CO)_3I$ (6)

A solution of Cp^sH (0.35 g, 1.14 mmol) in toluene (20 ml) is added to a solution of $Mo(CO)_3(NCMe)_3$ (0.4 g, 1.14 mmol) in toluene (20 ml) at r.t. The reaction mixture is stirred for 2 h and the resulting orange solution is filtered through Celite. The filtrate is concentrated to dryness and the residue is dissolved in dichloromethane (50 ml). Solid CHI_3 (0.45 g, 1.14 mmol) is added to the dichloromethane solution and the colour immediately turns to a deep red. The reaction mixture is stirred for a further 30 min to ensure completion of the reaction and the solvent is removed under vacuum. The title compound **6** is isolated as a red solid. Yield 0.51 g (68%). Anal. Calc. for $C_{22}H_{31}MoO_7SiI$: C, 40.13; H, 4.75. Found: C, 39.84; H, 4.45%. ^{1}H-NMR ($CDCl_3$, 300 MHz, 25 °C δ ppm): 5.84 (d, 1H, H-1, $J_{1,2} = 3.0$ Hz), 5.63–5.61 (m, 1H, Cp^s), 5.55–5.53 (m, 1H, Cp^s), 5.32–5.31 (m, 2H, Cp^s), 4.31 (d, 1H, H-2), 4.13–4.09 (m, 1H, H-4), 4.04 (d, 1H, H-3, $J_{3,4} = 3.0$ Hz), 2.84 (dd, 1H, H-5a, $J_{5a,5b} = 15.0$ Hz, $J_{5a,4} = 9.0$ Hz), 2.53 (dd, 1H, H-5b, $J_{5b,4} = 3.0$ Hz), 1.41 (s, 3H, CH_3-isop), 1.24 (s, 3H, CH_3-isop), 0.85 (s, 9H, *tert*-Bu), 0.08 (s, 3H, CH_3-silyl), 0.07 (s, 3H, CH_3-silyl). ^{13}C-NMR ($CDCl_3$, 300 MHz, 25 °C δ ppm): 237.2, 220.8 (s, CO); 116.5 (s, Cq, isop); 111.5 (s, Cq, Cp^s); 104.8 (s, CH, C1); 95.7 (s, CH, Cp^s); 90.8 (s, CH, Cp^s); 85.5, 81.3, 76.7 (s, CH, C2–C4); 29.2 (s, CH_2, C5); 26.7 (s, CH_3-isop); 26.2 (s, CH_3-isop); 25.7 (s, *tert*-Bu); 18.0 (s, Cq, *tert*-Bu); −4.7 (s, CH_3-silyl); −5.0 (s, CH_3-silyl). Selected IR (KBr, cm^{-1}): 2036, 1963, 1884, vs, ν(CO).

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