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# Synthesis and X-ray crystal structure of 2-(methoxymethyl) phenyldicyclopentadienyltitanium(III)

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#### Abstract

Reaction of bis(2-methoxymethylphenyl)magnesium (1a) with Cp<sub>2</sub>TiCl<sub>2</sub> in THF affords Cp<sub>2</sub>Ti[2-CH<sub>3</sub>OCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>] (3) and 2,2'bis(methoxymethyl)biphenyl in high yield. The crystal structure of 3 has been determined and is the first structure of a Ti(III) compound in which the Ti atom is intramolecularly coordinated by oxygen. Two chemically identical and conformationally only slightly different residues were found to be present in the unit cell of 3. Compound C<sub>18</sub>H<sub>19</sub>OTi (3) crystallizes in the monoclinic space group Pc (no. 7) with a = 13.0248(8), b = 7.7769(5), c = 14.8491(7)Å,  $\beta = 106.448(4)^\circ$ , V = 1442.55(15)Å<sup>3</sup>, Z = 4. The structure refinement converged to R1 = 0.0478 for 4891  $F_0$ / $4\sigma(F_0)$  and wR2 = 0.0968 for all 6600 unique data, S = 1.010. The structure shows a pseudotetrahedral coordination around the Ti atom by two  $\eta^5$ -cyclopentadienyl groups, one carbon of the aryl ligand (Ti-C 2.19 Å) and an oxygen atom of the methoxymethyl group. The Ti-O distance of 2.20 Å is 1.48 Å shorter than the expected van der Waals distance, which indicates a strong interaction between Ti and O. Reaction of bis(2-N, N-dimethylaminomethylphenyl)magnesium (1b) and Cp<sub>2</sub>TiCl<sub>2</sub> affords in a similar fashion Cp<sub>2</sub>Ti[2-(CH<sub>3</sub>)<sub>2</sub>NCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>] (4) and 2,2'-bis(N, N-dimethylaminomethyl)biphenyl.

Keywords: Titanium(III); Intramolecular coordination; Crystal structure; Chelate; Group 4

#### **1. Introduction**

Compounds of the type  $Cp_2 TiR$  (R = alkyl or aryl) have been shown to be unstable unless complexation by a donor molecule containing nitrogen or oxygen occurs [1]. However, the stability is enhanced significantly when the active site is blocked sterically [2] or by intramolecular coordination [3].

A general method for the synthesis of Cp<sub>2</sub>TiR (R = alkyl, aryl) compounds consists of the reaction between [ClTiCp<sub>2</sub>]<sub>2</sub> and a Grignard or organolithium reagent. Most of the titanium(III) compounds have been synthesized in this manner. However, titanium(III) compounds can also be obtained from Cp<sub>2</sub>TiCl<sub>2</sub>. For example, the reaction of Cp<sub>2</sub>TiCl<sub>2</sub> with two equivalents of allylmagnesium chloride furnished  $\pi$ -allyltitanocene and propene [4]. Similarly, reaction of  $Cp_2TiCl_2$  with RLi (R = CHPh<sub>2</sub> or CH(SiMe<sub>3</sub>)<sub>2</sub>) gave the corresponding  $Cp_2TiR$  compounds [5]. Thermolysis of Ti(1V) compounds can also give Ti(III) compounds; for example, the thermolysis of  $Cp_2TiMe_2$  resulted in the formation of  $Cp_2TiMe$  and of a methyl radical [6,7]. So far, none of the above-mentioned methods starting from  $Cp_2TiCl_2$  has yielded an intramolecularly coordinated Ti(III) compound.

We were interested in the synthesis of titanocene derivatives with the potential of intramolecular coordination of the formula  $Cp_2Ti(2-XCH_2C_6H_4)_2$  (X = MeO (2a), Me<sub>2</sub>N (2b)) for two reasons. In the first place, intramolecular coordination of oxygen or nitrogen might enhance the stability of the titanium(IV) compound in comparison with the unsubstituted diphenyltitanocene. Secondly, we wanted to investigate whether treatment of (2a,b) with PMe<sub>3</sub> would induce migration of one of the aryl rings to one of the cyclopentadienyl rings. However, although compounds 2a and 2b could be

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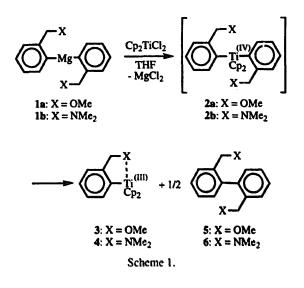
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synthesized at low temperature (-20°C), they started to decompose at this temperature with tormation of the more stable titanium(III) compounds 3 and 4 respectively. Here, we report the synthesis of two Ti(III) compounds 3 and 4 by reaction of  $Cp_2TiCl_2$  with (2-XCH<sub>2</sub>C<sub>6</sub>H<sub>4</sub>)<sub>2</sub>Mg (X = MeO, Me<sub>2</sub>N).

#### 2. Results and discussion

## 2.1. Synthesis of $Cp_2Ti[2-CH_3OCH_2C_6H_4]$ (3) and $Cp_2Ti[2-(CH_3)_2NCH_2C_6H_4]$ (4)

Treatment of a THF solution of bis(2-methoxymethylphenyl)magnesium (1a) with  $Cp_2TiCl_2$  at -20°C furnished the corresponding Ti(IV) complex 2a, which, upon warming to room temperature, quantitatively decomposed to give the Ti(III) compound 3 and the corresponding 2,2'-disubstituted biphenyl (5) (Scheme 1). The reaction mixture obtained from 1a and  $Cp_2TiCl_2$  at  $-20^{\circ}C$  was evaporated to dryness at -20°C; the residue was dissolved in C<sub>6</sub>D<sub>6</sub>, allowed to warm to room temperature and immediately cooled to 5°C, and the <sup>1</sup>H NMR spectrum was measured. It showed the presence of 2a and 5 only. Thus, the formation of compound 3 proceeds via the corresponding Ti(IV) compound, which undergoes radical cleavage to furnish 3 and 5. In a similar fashion, compound 4 was formed from bis(2-N,N-dimethylaminomethylphenyl)magnesium (1b); it has been synthesized previously by Teuben and coworkers by reaction of (2- $Me_2 NCH_2C_6H_4$ )Li with  $[Cp_2TiCi]_2$  in 67% yield [8]. Recrystallization of the Ti(III) compounds from n-pentane afforded a crystalline green 3 and a purple 4, both in good yield. The X-ray crystal structure of compound 4 has been described [9].



Complex 3 is sensitive to air but can be stored for months in pentane solution at room temperature in evacuated glassware without noticeable decomposition. The complex is paramagnetic; although it is known that several Ti(III) compounds are reactive towards isocyanides [10] and nitrogen [2], 3 does not react with tert-BuN≡C or nitrogen. However, treatment of the reaction mixture containing 3 with gaseous HCl furnished almost quantitatively methoxymethylbenzene and 2.2'-bis(methoxymethyl)biphenyl (5) in the expected ratio 2:1. For the characterization of 3 by NMR spectroscopic techniques, a corresponding diamagnetic Ti(IV) compound would have been desirable. Therefore, 3 was reacted with PbCl<sub>2</sub>, which is a well-known oxidizing reagent for this purpose [11]. However, a diamagnetic compound was not obtained.

The mass spectra of 3 and 4 using electron impact (EI) as ionization technique did not show any signals at

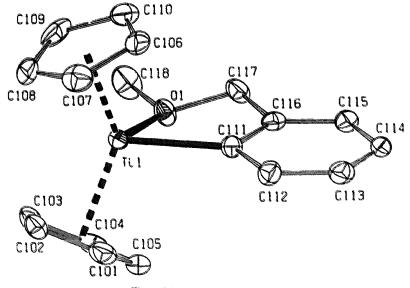


Fig. 1. Molecular structure of 3.

higher m/z values than those of the molecular ion cluster of the monomers, suggesting that 3 and 4 are monomeric in the gas phase.

#### 2.2. X-ray crystal structure determination of (3)

Crystallization of 3 from *n*-pentane yielded crystals suitable for an X-ray crystal structure determination; the molecular structure is depicted in Fig. 1, while selected bond lengths and angles are listed in Table 1. It is the first crystal structure of a Ti(III) compound in which the Ti atom is intramolecularly coordinated by oxygen. The unit cell consists of two chemically identical, crystallographically independent, and conformationally only units different monomeric of slightly  $Cp_2Ti[2(CH_3OCH_2C_6H_4];$  only one of them is depicted in Fig. 1. The Ti atom is pseudotetrahedrally coordinated by one carbon atom (C(111)) of the aryl group, the oxygen atom of the methoxy group and the two  $n^{5}$ -cyclopentadienyl groups. The planes through Cp(1)(centroid)-Ti-Cp(2)(centroid) and through O-Ti-C(111) are nearly perpendicular (89.66°). The Ti-Odistance of 2.20 Å is 1.48 Å shorter than the sum of the van der Waals radii (r(Ti) ≈ 2.16 Å [12], r(O) = 1.52 Å [12];  $\Sigma = 3.68$  Å) indicating a strong interaction between Ti and O. (Van der Waals radii for N and O were taken from Ref. [12], and for Ti they were estimated from the covalent radius  $r_{vdW} = 1.36 + 0.8$ .) This interaction is even stronger than that between Ti and N in 4, where the Ti-N distance (2.46(2)Å) [9] is only 1.15 Å shorter than the sum of the van der Waals radii (r(N) =1.55 Å [12]). The angle O-Ti-C(111) of 73.9° is practically identical with the corresponding angle N-Ti-C in complex 4 (73.1°).

Table 1 Bond lengths (Å) and bond angles (deg) for 3 (E = O) and corresponding values for 4 (E = N) [9] with e.s.d.s in parentheses<sup>a,b</sup>

	3(n=1)	<b>3</b> (n ≈ 2)	4
Ti-E	2.200(3)	2.209(3)	2.475
Ti-C(n11)	2.193(3)	2.197(4)	2.233
Ti-Cpl	2.0644(19)	2.062(2)	2.08
Ti-Cp2	2.068 (2)	2.064(2)	2.09
C(n 7) = O	1.442(4)	1.448(4)	1.466
C(n16) - C(n17)	1.501 (6)	1.496(5)	1.566
O-Ti-C(nl1)	73.89(13)	73.62(13)	72.95
C(n 2)-C(n 1)-Ti	129.0(3)	128.2(3)	127.10
C(n16)-C(n17)-O	108.1(3)	107.9(3)	111.72
C(n17) - O - C(n18)	111.6(3)	111.5(3)	106.98
Cp1-Ti-Cp2	135.32(3)	135.28(3)	131
Ti-C(n11)-C(n16)-C(n15)	1.2(7)	0.0(7)	2.7(2)
E-C(n 7)-C(n 6)-C(n 1)	15.6(5)	18.4(4)	36.7(8)
C(n12)-C(n11)-C(16)-C(n17)	1.4(6)	2.8(7)	1.1(5)

<sup>a</sup> Cp denotes Cp-centroid. <sup>b</sup> n is the molecule number.

#### **3. Experimental details**

#### 3.1. General

All manipulations were carried out in fully sealed glassware without ground joints or stopcocks using high vacuum techniques. Solvents were dried by distillation from liquid Na-K alloy (THF, *n*-pentane) after predrying on NaOH (THF). The organomagnesium compounds were prepared as described in the literature [13].

GC-MS analyses were performed on a Hewlett-Packard 5970 Mass Selective Detector, equipped with a Hewlett-Packard 5890 gas chromatograph. GC analyses were performed on an Intersmat gas chromatograph GC 120 equipped with a glass column 10% OV-101 chromosorb WHP 80/100. High resolution mass spectrometry measurements were performed on a Finnigan MAT 90. NMR spectra in  $C_6D_6$  were measured on a Bruker AC 200 spectrometer; ESR spectra were measured in  $C_6D_6$  on a Bruker ESP 300 spectrometer. The elemental analysis was performed at the Mikroanalytical Laboratory, Rijksuniversiteit Groningen, Groningen, The Netherlands.

#### 3.2. Preparation of $Cp_2Ti[2-CH_3OCH_2C_6H_4]$ (3)

A solution of  $[2-CH_3OCH_2C_6H_4]_2$  Mg (1a) (75.2 mg, 0.283 mmol) in THF (6.2 ml) was added to  $Cp_2TiCl_2$ (71 mg, 0.285 mmol) at -20 °C. The reaction mixture immediately turned orange. After 10 min of stirring at -20°C, the reaction mixture turned orange-red. After 6 days of stirring at -20 °C the solvent was evaporated in vacuo from the orange-red reaction mixture at -20 °C. The 'H NMR spectra of a sample of the residue showed the presence of compound 2a. The remaining residue was dissolved in *n*-pentane (10 ml) and warmed to room temperature. The orange-red solution turned yellow-green. By cooling this solution, green crystals of 3 were obtained. In a second, identical experiment, the n-pentane solution was hydrolysed and worked up as usual; methoxymethylbenzene and 2.2'bis(methoxymethyl)biphenyl (5) were obtained in a ratio 2:1 (based on GC analysis). After evaporation of the solvent, the residue was subjected to sublimation which yielded 5 as a white solid (32 mg, 93%); 'H NMR (200 MHz,  $C_6 D_6$ , Ref.  $C_6 D_5 H = 7.17 \text{ ppm}$ )  $\delta$  7.68 (d,  ${}^{3}J(HH) = 7.2 Hz, 2H, ArH), 7.20-7.11 (m, 6H, ArH),$ 4.18 (s, 4H, CH<sub>2</sub>), 3.00 (s, 6H, CH<sub>3</sub>). HRMS: calculated for C<sub>15</sub>H<sub>14</sub>O: 210.1045; observed: 210.1048.

**2a**: <sup>1</sup>H NMR (200 MHz, C<sub>6</sub>D<sub>6</sub>, Ref. C<sub>6</sub>D<sub>5</sub>H = 7.17 ppm)  $\delta$  7.60–7.00 (m, 8H, Ar-H), 5.97 (s, 10H, C<sub>p</sub>), 4.18 (s, 4H, OCH<sub>2</sub>), 3.17 (s, 6H, OCH<sub>3</sub>).

3: m.p.:  $105-107 \,^{\circ}$ C. ESR (C<sub>6</sub>D<sub>6</sub>): g = 1.9869; line width 4 G. HRMS: calculated for C<sub>18</sub>H<sub>19</sub>O<sup>48</sup>Ti: 299.0915; observed: 299.0915. Mass spectrum (EI) m/z(rel. int.) 299 (37, M<sup>+</sup>), 267 (43), 266 (31), 265 (100), 241 (12), 179 (22), 178 (35), 144 (18), 129 (33), 128 (12), 122 (10), 121 (10), 91 (21), 65 (11). Anal. Found: C, 72.36; H, 6.31.  $C_{18}H_{19}O^{48}Ti$ . Calc.: C, 72.35; H, 6.40.

#### 3.3. Preparation of $Cp_2Ti[2-(CH_3)_2NCH_2C_6H_4]$ (4)

In a similar experiment, a solution of  $[2-(CH_3)_2NCH_2C_6H_4]_2Mg$  (1b) (45 mg, 0.154 mmol) in THF (10 ml) was added to  $Cp_2TiCl_2$  (35 mg, 0.14 mmol) at -20 °C. The reaction mixture immediately turned red. After stirring for one night at -20 °C, the reaction mixture had turned orange and the solvent was evaporated in vacuo at room temperature. The residue was extracted with *n*-pentane (10 ml); by cooling the solution, purple crystals of 4 were obtained. The *n*-pentane solution was hydrolysed and worked up as usual; dimethylaminomethylbenzene and 2,2'-bis(dimethyl-

 Table 2

 Crystallographic data for compound 3

 Crystal data

 Formula
 C<sub>18</sub>H<sub>19</sub>OTi

 Molecular weight
 299 23

Formula	C <sub>18</sub> H <sub>19</sub> OTi
Molecular weight	299.23
Crystal system	monoclinic
Space group	<i>Pc</i> (No. 7)
a (Å)	13.0248(8)
b (Å)	7.7769(5)
c (Å)	14.8491(7)
β (deg)	106.448(4)
V (Å3)	1442.55(15)
D <sub>eale</sub> (g cm = 3)	1.38
Z	4
F(000)	628
μ (cm <sup>=1</sup> )	5.9 (MoKa)
Crystal size (mm <sup>3</sup> )	$0.50 \times 0.25 \times 0.15$
Data collection	
<i>T</i> (K)	150
Osera-range	9.8-13.9
$\theta_{min},  \theta_{max}  (deg)$	1.6, 27.5
Wavelength (Å)	0.71073 (graphite
	monochromator)
Scan type	0
X-ray exposure time (h)	13.9
Linear decay (%)	2
Reference reflection	216, 423, 312
Data set	- 16:16, 0:10, - 19:19
Total data	6915
Total unique data	6600
Refinement	<b>A</b> # <b>A</b>
No. of refined parameters Final R1, no. of data <sup>a</sup>	363
Final wR2, no. of data <sup>b</sup>	$0.0478, 4891 F_o > 4\sigma(F_o)$
S I MAR WAR, NO. OI URR.	0.0968, 6600
Flack parameter	1.010 0.02(3)
W <sup>-10</sup>	$\sigma^{2}(F^{2}) + (0.0392P)^{2} + 0.11P$
$(\Delta/\sigma)_{av}, (\Delta/\sigma)_{max}$	0.000, -0.001
Min., max. residual	-0.31, 0.33
density (e Å = 3)	- 0.31, 0.33
wenony (CA )	

<sup>a</sup>  $R_1 = \sum ||F_0| - |F_0|| / \sum |F_0|;$  <sup>b</sup>  $wR_2 = \{\sum ||w(F_0^2 - F_0^2)^2] / \sum ||w(F_0^2)^2|\}^{1/2};$  <sup>c</sup>  $P = (Max(F_0^2, 0) + 2F_0^2) / 3.$ 

Table 3

Final coordinates and equivalent isotropic thermal parameters of the nonhydrogen atoms for compound 3

. Only of	ogen atoms re	or compound 3		
Atom	x	у	z	$U_{eq}$ (Å <sup>2</sup> )
Ti(1)	0.41296(4)	0.44528(9)	0.24693(4)	0.0160(2)
<b>O(1)</b>	0.2590(2)	0.5632(4)	0.1717(2)	0.0219(8)
C(101)	0.4435(3)	0.1953(5)	0.3396(3)	0.0258(12)
C(102)	0.4797(3)	0.3334(5)	0.4012(3)	0.0238(12)
C(103)	0.3903(3)	0.4354(6)	0.4012(2)	0.0273(12)
C(104)	0.2995(3)	0.3575(6)	0.3424(3)	0.0282(14)
C(105)	0.3313(3)	0.2101(5)	0.3039(3)	0.0264(12)
C(106)	0.5398(3)	0.5133(5)	0.1617(3)	0.0253(12)
C(107)	0.5976(3)	0.4861(6)	0.2558(3)	0.0304(14)
C(108)	0.5683(3)	0.6151(6)	0.3098(3)	0.0325(16)
C(109)	0.4920(4)	0.7192(6)	0.2494(3)	0.0340(16)
C(110)	0.4747(3)	0.6574(5)	0.1577(3)	0.0285(12)
C(111)	0.3500(3)	0.2925(5)	0.1190(2)	0.0178(12)
C(112)	0.3887(3)	0.1358(5)	0.0930(3)	0.0220(12)
C(113)	0.3435(3)	0.0546(5)	0.0081(2)	0.0233 (12)
C(114)	0.2538(3)	0.1256(5)	-0.0555(3)	0.0225(12)
C(115)	0.2118(3)	0.2780(5)	-0.0333(3)	0.0211 (12)
C(116)	0.2597(3)	0.3589(5)	0.0523(3)	0.0185(12)
C(117)	0.2148(3)	0.5273(5)	0.0730(2)	0.0229(12)
C(118)	0.2249(4)	0.7260(7)	0.1962(3)	0.0342(17)
Ti(2)	0.83234(4)	0.04981(10)	0.60601(4)	0.0170(2)
O(2)	0.9891 (2)	- 0.0583(4)	0.6059(2)	0.0223(8)
C(201)	0.8022(3)	0.3049(5)	0.6793(3)	0.0268(12)
C(202)	0.7654(3)	0.1709(6)	0.7247(3)	0.0279(12)
C(203)	0.8533(3)	0.0694(6)	0.7714(3)	0.0326(12)
C(204)	0.9441(3)	0.1429(6)	0.7564(3)	0.0309(14)
C(205)	0.9142(3)	0.2876(6)	0.6995(3)	0.0299(14)
C(206)	0.7000(3)	- 0.0247(6)	0.4631(3)	0.0348(14)
C(207)	0.6472(3)	- 0.0097(6)	0.5322(3)	0.0335(16)
C(208)	0.6866(3)	- 0.1384(6)	0.5995(3)	0.0348(16)
C(209)	0.7640(4)	- 0.2328(6)	0.5719(4)	0.0414(18)
C(210)	0.7721(4)	- 0.1616(6)	0.4875(3)	0.0396(17)
C(211)	0.8886(3)	0.2026(5)	0.5049(2)	0.0176(12)
C(212)	0.8440(3)	0.3552(5)	0.4584(3)	0.0235(12)
C(213)	0.8875(3)	0.4393(5)	0.3948(2)	0.0274(12)
C(214)	0.9797(3)	0.3753(5)	0.3765(3)	0.0258(12)
C(215)	1.0257(3)	0.2260(5)	0.4205(2)	0.0230(12)
C(216)	0.9795(3)	0.1434(5)	0.4833(2)	0.0181(12)
C(217)	1.0286(3)	-0.0221(5)	0.5260(2)	0.0216(12)
C(218)	1.0280(4)	- 0.2190(6)	0.6481(3)	0.0325(17)

 $U_{\rm eq} = 1/3$  of the trace of the orthogonalized  $\mathcal{C}$ .

aminomethyl)biphenyl (6) were obtained in a ratio 2:1 (based on GC).

6: the 'H NMR spectrum was in accordance with that reported [14].

4: ESR ( $C_6D_6$ ): g = 1.9862; line width 3.5 G. HRMS: calculated for  $C_{19}H_{22}N^{48}Ti$ : 312.1232; observed: 312.1231. Mass spectrum (EI) m/z (rel. int.) 312 (55, M<sup>+-</sup>), 265 (80), 204 (47), 134 (100, M<sup>+-</sup> – TiCp<sub>2</sub>), 91 (17), 58 (10).

### 3.4. X-ray structure determination of 3

Crystal data and details on data collection and refinement are presented in Table 2. Final coordinates and equivalent isotropic thermal parameters are given in Table 3.

A transparant, green crystal was cut to size, mounted on a Lindemann glass capillary, and subsequently transferred into the cold nitrogen stream on an Enraf-Nonius CAD4-T diffractometer on rotating anode. Accurate unit-cell parameters and an orientation matrix were determined from the setting angles of 25 reflections (SET4) [15]. Data were collected at 150K in  $\omega$  scan mode with scan angle  $\Delta \omega = 0.75 + 0.35 \tan \theta^{\circ}$ . Data were corrected for Lp effects, and for a linear decay of 2% for the three periodically measured reference reflections. The structure was solved by automated direct methods (SIR92 [16]). Refinement on  $F^2$  was carried out by full-matrix least squares techniques (SHELXL-93 [17]); no observance criterion was applied during refinement. All non-hydrogen atoms were refined with anisotropic thermal parameters. The hydrogen atoms were refined with a fixed isotropic thermal parameter amounting to 1.5 or 1.2 times the value of the equivalent isotropic thermal parameter of their carrier atoms, for the methyl hydrogen atoms, and the other hydrogen atoms respectively. Weights were optimized in the final refinement cycles. The unit-cell contains two symmetry-independent molecules, but no higher crystallographic symmetry could be detected with reduced-cell calculations [18]. Neutral atom scattering factors and anomalous dispersion corrections were taken from International Tables for Crystallography [19].

Geometrical calculations and illustrations were performed with PLATON [20]; all calculations were performed on a DEC5000/125. Further details of the crystal structure investigation may be obtained from one of the authors (A.L.S.).

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