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# Formation of [Cp<sub>2</sub>TiSbMe<sub>2</sub>]<sub>2</sub>, [Cp<sub>2</sub>TiSb(SiMe<sub>3</sub>)<sub>2</sub>]<sub>2</sub> and [Cp<sub>2</sub>TiCl]<sub>2</sub>·2Mes<sub>4</sub>Sb<sub>2</sub>

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### 1. Introduction

Recently we described the syntheses of the first titanocene complexes with organoantimony ligands:  $[Cp_2Ti(Sbdmp)_2]$  (dmp = 2,6-Mes<sub>2</sub>C<sub>6</sub>H<sub>3</sub>) [1],  $[Cp_2TiSbR_2]_2$  (R = Et),  $[(Cp_2Ti)_3(RSb)_3Sb]$  (R = 2-(Me<sub>2</sub>NCH<sub>2</sub>)C<sub>6</sub>H<sub>4</sub>) and  $[(Cp_2Ti)_5(RSb)_2Sb_7]$  (R = Me<sub>3</sub>SiCH<sub>2</sub>) [2] by reactions of the titanocene source  $[Cp_2Ti(btmsa)]$ , (btmsa = bis(trimethylsilyl)acetylene, Me<sub>3</sub>SiC=CSiMe<sub>3</sub>) with antimony compounds of the type RSbH<sub>2</sub> or R<sub>2</sub>SbSbR<sub>2</sub>. Very few related complexes of Zr and Hf including  $[Cp_2Zr(SbPh_2)_2]$  [3],  $[CpCp^*HfSbdmp]$  [4],  $[CpCp^*Hf(Cl)Sb(H)$  dmp] [5],  $[CpCp^*Hf(SbMes_2)_2]$  [6] are known. Analogous compounds with phosphorus or arsenic ligands have been studied more frequently and group 4 metal complexes with R<sub>3</sub>E, R<sub>2</sub>E, RE, R<sub>3</sub>E<sub>3</sub> (E = P, As) ligands have been synthesized [7–14].

We report here on reactions between  $[Cp_2Ti(btmsa)]$  and  $R_2SbSbR_2$  (R = Me,  $Me_3Si$ ) giving the novel Sb–Ti complexes  $[Cp_2TiSbMe_2]_2$  (**1**) and  $[Cp_2TiSb(SiMe_3)_2]_2$  (**2**) respectively and on the serendipitous formation of  $[Cp_2TiCl]_2 \cdot 2Mes_4Sb_2$  (**3**) from  $[Cp_2Ti(btmsa)]$  and  $Mes_2SbH$  containing NH<sub>4</sub>Cl traces.

## 2. Results and discussion

The distibanes  $R_4Sb_2$  ( $R = Me, Me_3Si$ ) react with [Cp<sub>2</sub>Ti(btmsa)] in benzene at room temperature with exchange of the btmsa ligand and formation of crystalline samples of [Cp<sub>2</sub>TiSbMe<sub>2</sub>]<sub>2</sub> (1) or

#### ABSTRACT

Reactions of  $[Cp_2Ti(btmsa)]$  (btmsa = bis(trimethylsilyl)acetylene) with R<sub>4</sub>Sb<sub>2</sub> (R = Me, Me<sub>3</sub>Si) give  $[Cp_2TiSbMe_2]_2$  (1) or  $[Cp_2TiSb(SiMe_3)_2]_2$  (2) respectively.  $[Cp_2TiCl]_2 \cdot 2Mes_4Sb_2$  (3) is serendipitously formed from  $[Cp_2Ti(btmsa)]$  and Mes<sub>2</sub>SbH containing NH<sub>4</sub>Cl traces.

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 $[Cp_2TiSb(SiMe_3)_2]_2$  (2) respectively. The reaction is described in equation (1). The first steps probably are the substitution of btmsa and the insertion of titanocene into the Sb–Sb bond of the distibane with intermediate formation of  $[Cp_2Ti(SbR_2)_2]$ . In the final step 1 and 2 are obtained by reaction with a second titanocene moiety. 1 is the only isolated product although the 1:1 M ratio of Me<sub>4</sub>Sb<sub>2</sub> and  $[Cp_2Ti(btmsa)]$  was applied. 1 and 2 can be viewed as being composed of  $Cp_2Ti^+$  and  $R_2Sb^-$  and therefore the reaction (1) is a redox process with oxidation of Ti(II) to Ti(III) and reduction of Sb (II) to Sb(I).

$$\begin{array}{rcl} 2[Cp_2Ti(btmsa)]+R_2SbSbR_2 & \rightarrow & [Cp_2TiSbR_2]_2+2 \ btmsa \\ & 1,2 \\ 1:R = Me \\ 2:R = Me_3Si \\ btmsa = Me_3SiC \equiv CSiMe_3 \end{array} \tag{1}$$

The analogous ethyl derivative [Cp<sub>2</sub>TiSbEt<sub>2</sub>]<sub>2</sub> was obtained in a similar way [2]. Also in presence of excess tetramethyldistibane we found no evidence for [Cp<sub>2</sub>Ti(SbMe<sub>2</sub>)<sub>2</sub>], a complex that might be formed not only initially by the insertion reaction of titanocene with tetramethyldistibane but also by addition of the distibane to **1**.

Related to compound **2** is the monomeric phosphorus complex  $[Cp_2TiP(SiMe_3)_2]$  that was formed from  $[Cp_2TiCl]_2$  and  $LiP(SiMe_3)_2$  [15]. It is remarkable that **2** is a dimer in the solid state whereas the analogous phosphorus complex is monomeric. This difference however reflects the steric situation. As a consequence of the increase of the atomic radii from phosphorus (1.1 Å) to antimony

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(1.4 Å) the protection by Me<sub>3</sub>Si groups is sufficient to prevent the phosphorus monomer from dimerization but not a [Cp<sub>2</sub>TiSb (SiMe<sub>3</sub>)<sub>2</sub>] monomer.

The new complexes are orange (1) or red (2) air sensitive compounds. Their characterization is based mainly on single crystal X-ray crystallography. Meaningful NMR spectra were not observed and mass spectra showed only ions corresponding to decomposition products. Elemental analyses were not carried out because of the sensitivity of the samples. 2 was isolated as crystalline solid only in reduced quantities. Nevertheless, it is possible that in solution the amount of 2 could have been larger but the identification of the compounds was hindered by its magnetic properties. The molecular structures of 1 and 2 are shown in the Figs. 1 and 2.

The structures of **1** and **2** consist of R<sub>2</sub>Sb units between two Cp<sub>2</sub>Ti groups forming a planar (SbTi)<sub>2</sub> heterocycle. The Sb atoms adopt a distorted tetrahedral geometry with wider Ti–Sb–Ti angles (**1**: 105.32(5), **2**: 103.384(13)°) and smaller C(2)–Sb(1)–C(1) 93.8 (6) or Si–Sb–Si (94.51(2)°) angles. Related structural parameters were reported for [Cp<sub>2</sub>TiSbR<sub>2</sub>]<sub>2</sub> (R = Et). The transannular Sb···Sb distances (**1**: 3.4991(11), **2**: 3.7145(4) Å) are shorter than the sum of the van-der-Waals radii of two antimony atoms [ $\Sigma$ r.v.d.W (Sb, Sb) = 4.4 Å]. The Sb–Ti-bond lengths in **2** (2.999(4) Å) are larger than in **1** (2.8800(17), 2.8890(18) Å) or [Cp<sub>2</sub>TiSbEt<sub>2</sub>]<sub>2</sub> (molecule 1: 2.8797(11), 2.8917(11); molecule 2: 2.9046(11), 2.9105(11) Å).

The inspection of the <sup>1</sup>H NMR spectra during the synthesis of **1** revealed that the signals of the starting materials [Cp<sub>2</sub>Ti(btmsa)] [ $\delta = 6.40$  (C<sub>5</sub>H<sub>5</sub>), 0.33 (CH<sub>3</sub>)] and Me<sub>4</sub>Sb<sub>2</sub> ( $\delta = 0.82$ ) decreased in intensity during the reaction whereas the signal for non coordinated btmsa ( $\delta = 0.14$ ) increased. However no new signals appeared in the spectra. Also when crystals of **1** or **2** were solved in C<sub>6</sub>D<sub>6</sub> no characteristic NMR signals were observed. This absence of NMR signals is not surprising because also the analogous ethyl derivative was found NMR silent and DFT calculations on [Cp<sub>2</sub>TiSbR<sub>2</sub>]<sub>2</sub> (R = Et) had shown that indeed the paramagnetic state with two unpaired electrons at the Ti(III) centers with d<sup>1</sup> configuration is lower in energy than the diamagnetic state with coupled electrons [2]. It can be concluded that **1** and **2** are paramagnetic at room temperature as well.

Mass spectra were measured from crystalline samples of **1** or **2** with the EI technique. The spectra showed no signals of the intact Sb–Ti complexes. Instead intense signals of the distibanes,  $(Me_4Sb_2, m/z = 304, M^+; (Me_3Si)_4Sb_2, m/z = 534, M^+)$  and of the

0.2

Sb1

Sb1

C2

C11

C1

**Fig. 1.** Thermal ellipsoid (20%) representation of **1**. The hydrogen atoms were omitted for clarity. Selected bond lengths (Å) and bond angles ( $^{\circ}$ ): Sb(1)–Ti(1) 2.881(2), Sb(1)–Ti(1'), 2.889(2), C(1)–Sb(1) 2.163(12), C(2)–Sb(1) 2.154(12), C(2)–Sb(1)–C(1) 94.3(7), Sb(1)–Ti(1)–Sb(1'), 74.66(5) Ti(1)–Sb(1)–Ti(1') 105.34(5).



**Fig. 2.** Thermal ellipsoid (20%) representation of **2**. The hydrogen atoms were omitted for clarity. Selected bond lengths (Å) and bond angles (°): Ti(1)–Sb(1) 2.9994(4), Ti(1')–Sb(1') 2.9927(5), Si(1)–Sb(1) 2.5842(6), Si(2)–Sb(1) 2.5824(6), Sb(1)···Sb(1') 3.7145(4), Ti(1)–Sb(1)–Ti(1') 103.384(13), Si(1)–Sb(1)–Si(2) 94.51(2), Si(1)–Sb(1)–Ti (1) 114.779(16), Si(2)–Sb(1)–Ti(1) 114.870(16), Sb(1)–Ti(1)–Sb(1') 76.616(13).

molecular ion of Cp<sub>2</sub>Ti (m/z = 178, 100%) were observed. The spectra contain also signals for Cp<sub>n</sub>Ti<sub>6</sub> (n = 4-8) clusters. These results indicate that **1** and **2** decompose under the conditions of mass spectrometry with the formation of the parent distibanes and titanocene monomers and oligomers.

In an attempt to synthesize analogues of **1** and **2** containing dimesitylantimony units we reacted  $[Cp_2Ti(btmsa)]$  with Mes<sub>2</sub>SbH (Mes = 2,4,6-Me<sub>3</sub>C<sub>6</sub>H<sub>2</sub>) in benzene at ambient temperature. After working up the product mixture several crystals of  $[(Cp_2TiCl)_2]\cdot 2$  (Mes<sub>2</sub>Sb)<sub>2</sub> (**3**) were obtained. The source of the chlorine atoms is probably ammonium chloride used during the preparation of Mes<sub>2</sub>SbH. In related reactions (Ph<sub>2</sub>P)<sub>2</sub> and  $[(Cp_2TiCl)_2]$  were formed from Cp<sub>2</sub>Ti(btmsa) and Ph<sub>2</sub>PCI [13]. The crystal structure of **3** was determined by single crystal X-ray diffraction. The structure is shown in Fig. 3.

Crystals of **3** contain the complex  $[(Cp_2TiCl)_2]$  and tetramesityldistibane in the ratio 1: 2. The structures of the components of **3** were determined independently several years ago. The geometrical parameters of **3** and the pure compounds  $[(Cp_2TiCl)_2]$  [15] and  $(Mes_2Sb)_2$  [16,17] are similar. Future work will concentrate on obtaining the analogue of **1** and **2** containing dimesitylantimony unit by the reaction of pure Mes\_2SbH with [Cp\_2Ti(btmsa)].

#### 3. Conclusion

C12

C11'

C5

Reactions between  $[Cp_2Ti(btmsa)]$  and substituted distibanes offer a feasible access to dimeric complexes between titanocene and diorganoantimony units. In contrast distibane formation is favoured in reactions between  $Cp_2Ti(btmsa)$  and  $Mes_2SbH$  with NH<sub>4</sub>Cl traces.

#### 4. Experimental

Crystals suitable for X-ray diffraction of **1–3** were grown from concentrated benzene solutions in case of **2** and **3** or deuterated benzene solution in case of **1** Table 1. Data were collected at 173 (2) K on a Siemens P4 diffractometer using a 0.71073 Å MoK $\alpha$  radiation and corrected for absorption effects using DIFABS [18]. The structures were solved by direct or Patterson methods [19]. Structure solutions and refinements were performed using WinGX software package [20]. The representations of the X-ray structures



**Fig. 3.** Thermal ellipsoid (20%) representation of **3**. The hydrogen atoms were omitted for clarity. Selected bond lengths (Å) and bond angles (°): Sb(1)–C(10) 2.174(8), Ti(1)–Cl(1)–Ti(1') 99.13(12), Sb(1)–C(1) 2.180(9), C(10)–Sb(1)–C(1) 97.3(3), Sb(2)–C(28) 2.203(9), C(19)–Sb(2)–C(28) 99.3(3), Sb(2)–C(19) 2.199(9), C(1)–Sb(1)–Sb(2) 94.9(2), Sb(1)–Sb(2) 2.8589(8), C(10)–Sb(1)–Sb(2) 108.9(2), Ti(1)–Cl(1) 2.511(4) C(19)–Sb(2)–Sb(1) 106.7(2), C(28)–Sb(2)–Sb(1) 89.8(2).

Table 1	
X-ray diffraction data and structure refinement details for <b>1–3</b> .	

Compound	1	2	3
Empirical formula	C24H32Sb2Ti2	C16H28SbSi2Ti	C46H54ClSb2Ti
Formula weight	659.80	446.21	933.74
Crystal system	Orthorhombic	Triclinic	Triclinic
Space group	Pbca	P -1	P -1
a (Å)	13.8496(16)	9.8862(7)	10.3111(14)
b (Å)	9.725(3)	10.4965(11)	14.1199(15)
<i>c</i> (Å)	17.397(3)	11.3165(9)	15.9629(14)
α (°)	90	86.724(8)	67.762(7)
β(°)	90	65.983(6)	72.249(9)
γ(°)	90	66.641(6)	76.945(10)
Volume (Å <sup>3</sup> )	2343.2(8)	977.10(15)	2032.6(4)
Z	4	2	2
Calculated density (Mg/m <sup>3</sup> )	1.87	1.517	1.526
Absorption coefficient (mm <sup>-1</sup> )	2.946	1.904	1.608
F(000)	1288	450	942
Crystal size (mm <sup>3</sup> )	$0.4 \times 0.1 \times 0.05$	$0.4\times0.3\times0.1$	$0.5\times0.15\times0.1$
θ range for data collection (°)	2.34 to 22.5	2.52 to 27.50	2.75 to 25.00
Index ranges (h, k, l)	±14,±10,±18	±11,-13/12, -14/13	$\pm 12, \pm 15, \pm 18$
Reflections	6734/1535	8876/4423	8405/6995
collected/unique	[0.1097]	[0.0227]	[0.0243]
[R(int)]			
Completeness to $\theta$ [ $\theta$ ] (%)	99.9 [22.5]	98.3 [27.5]	97.7 [25.0]
Max. and min.	0.8667 and	0.8324 and	0.8558 and
transmission	0.2322	0.5164	0.5003
Data/restraints/	1535/28/130	4423/0/187	6995/30 486
parameters			
Goodness-of-fit on F <sup>2</sup>	1.109	1.056	1.034
Final R indices $(I > 2\sigma(I))$	0.0545, 0.1065	0.0193, 0.0478	0.0610, 0.1561
$(K_1, WK_2)$ R indices (all data)	0.0909.0.1165	0.0217 0.0497	0 0002 0 1742
(P = wP)	0.0000,0.1100	0.0217, 0.0487	0.0902, 0.1743
(A1, WA2)	0.942 and	0.451 and	1 120 and
nesk and	0.642	0.324	1 396
hole (eÅ <sup>-3</sup> )	-0.042	-0.324	-1,300

were created using the Diamond software package. The starting materials  $R_4Sb_2$ , R = Me [21],  $Me_3Si$  [22], and  $Cp_2Ti(btmsa)$  [23] were prepared as described in the literature. Mes<sub>2</sub>SbH was prepared by the reaction of Mes<sub>3</sub>Sb with excess Li wire, followed by the protonation of the formed antimonide with NH<sub>4</sub>Cl, similarly to the method described by Cowley et al. [24].

- 4.1. Synthesis of  $[Cp_2TiSbMe_2]_2$  (1)
- a) 0.17 g (0.55 mmol)  $Me_4Sb_2$  were added to a solution of 0.20 g (0.57 mmol) [Cp<sub>2</sub>Ti(btmsa)] (btmsa =  $Me_3SiC_2SiMe_3$ ) in  $C_6D_6$ . The resulting brown mixture was kept at room temperature in a sealed NMR tube for 12 h when several orange crystals of **1** (m.p. dec. 90 °C) suitable for X-ray crystallography formed.
- b) 0.26 g (0.86 mmol) Me<sub>4</sub>Sb<sub>2</sub> were added to a solution of [Cp<sub>2</sub>Ti (btmsa)] (0.30 g, 0.86 mmol) in benzene. The resulting brown mixture was stirred for 4 h at room temperature and then the solvent was removed under vacuum and 0.16 g (45%) **1** remained as red-brown powder. MS (EI, 70 eV): *m/z* (relative intensity, %) 161 °C, 304 (100) [Me<sub>4</sub>Sb<sub>2</sub>], 289 (60) [Me<sub>3</sub>Sb<sub>2</sub>], 259 (42) [MeSb<sub>2</sub>], 151 (80) [Me<sub>2</sub>Sb]; 296 °C, 178 (100) [Cp<sub>2</sub>Ti], 66 (21) [CpH].

#### 4.2. Synthesis of $[Cp_2TiSb(SiMe_3)_2]_2$ (2)

A solution of 0.70 g (1.3 mmol)  $R_4Sb_2$  (R = Me<sub>3</sub>Si) in 5 mL benzene was added to 0.90 g (2.8 mmol) [Cp<sub>2</sub>Ti(btmsa)] in 10 mL benzene and the mixture was stirred at room temperature for 0.5 h. All the volatile components were removed at reduced pressure and the remaining oil was dissolved again in benzene. The mixture was stored for several days at -10 °C and very few red crystals suitable for X-ray crystallography formed. MS (EI, 70 eV): m/z (relative intensity, %) 79 °C, 534 (40), [R<sub>4</sub>Sb<sub>2</sub>], 73 (100) [R] (R = Me<sub>3</sub>Si); 412 °C, 807 (100) [Cp<sub>8</sub>Ti<sub>6</sub>], 741 (34). 676 (12), 611 (22), 544 (23), 480 (10), 178 (46) [Cp<sub>2</sub>Ti], 66 (60), 65 (60) [Cp].

## 4.3. Synthesis of [(Cp<sub>2</sub>TiCl)<sub>2</sub>]·2(Mes<sub>2</sub>Sb)<sub>2</sub> (3)

A solution of 0.56 g (1.6 mmol)  $Cp_2Ti(btmsa)$  in 10 mL benzene was added to 0.58 g (1.6 mmol)  $Mes_2SbH$  containing  $NH_4CI$  traces in 10 mL benzene. The mixture was stirred for 1 h and kept at ambient

temperature for 12 h. Thereafter the solvent was removed and the remaining viscous liquid was dissolved again in benzene. After 1 month at room temperature several yellow crystals of **3** formed. <sup>1</sup>H NMR (C<sub>6</sub>D<sub>6</sub>): Mes<sub>4</sub>Sb<sub>2</sub>, 2.06 (s, 3*H*, *p*-C*H*<sub>3</sub>), 2.45 (s, 6*H*, *o*-C*H*<sub>3</sub>), 6.65 (s, 2*H*, C<sub>6</sub>H<sub>2</sub>). MS (EI, 70 eV) 720 (5) [Mes<sub>4</sub>Sb<sub>2</sub>]<sup>+</sup>, 359 (20) [Mes<sub>2</sub>Sb]<sup>+</sup>, 213 (60) [Cp<sub>2</sub>TiCl]<sup>+</sup>, 148 (60) [CpTiCl]<sup>+</sup>, 119 (100) [Mes].

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## Appendix A. Supplementary material

CCDC 782141–782143 contain the supplementary crystallographic data for **1–3**. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc. cam.ac.uk/data\_request/cif.

Supplementary information associated with this article can be found in the online version at doi:10.1016/j.jorganchem.2010.09.007.

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