

Synthesis of $(\text{Me}_3\text{SiCH}_2\text{Sb})_n$ and reactions with ditellurides: crystal structure of $[\text{Cp}_2\text{Co}][\text{Me}_3\text{SiCH}_2\text{SbCl}_3]^\star$

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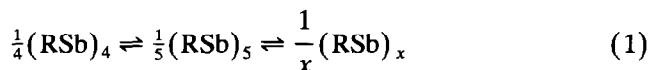
Abstract

Substitution of phenyl groups in Ph_2SbR (**1**) ($\text{R} = \text{Me}_3\text{SiCH}_2$) with chlorine atoms leads to RSbCl_2 (**2**). Reduction of **2** with magnesium in tetrahydrofuran gives red crystals of $(\text{RSb})_n$ (**3**) and a black solid containing the polymer $(\text{RSb})_x$. Solutions of **3** in C_6D_6 are yellow. They consist of an equilibrium mixture of $(\text{RSb})_5$ and $(\text{RSb})_4$. The reaction between **2** and cobaltocene in tetrahydrofuran or C_6D_6 leads to the polymer $(\text{RSb})_x$ and to the salt $[\text{CoCp}_2]^+[\text{RSbCl}_3]^-$ (**4**). The structure of **4** has been determined by single-crystal X-ray diffraction. It contains a centrosymmetric double-chlorine-bridged dimeric anion. The ditellurostibane $\text{RSb}(\text{Te}-p\text{-Tol})_2$ (**5**) has been obtained with a quantitative yield through a complete reaction between **3** and $(p\text{-TolTe})_2$. Compound **5** has been detected in the product mixtures resulting from reactions of polymeric $(\text{RSb})_x$ with $(p\text{-TolTe})_2$ or by reducing $p\text{-TolTeCl}_3$ and RSbCl_2 with magnesium in tetrahydrofuran.

Keywords: Antimony; Tellurium; X-ray structure

1. Introduction

Cyclostibanes $(\text{RSb})_n$ with n -alkyl substituents ($\text{R} = \text{Et}$, $n\text{-Pr}$ or $n\text{-Bu}$) have been reported [1] and investigated in relation to their structure and their thermochromic behaviour [2]. The ^1H NMR spectra reveal equilibria in solution between four- and five-membered homocycles, which reversibly polymerize to form chain compounds $(\text{RSb})_x$ in the absence of solvent [3]:



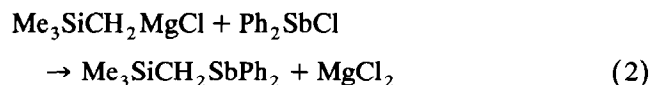
Both the homocycles and the polymers react with ditellurides [4] with formation of ditellurostibanes.

In order to enrich this class of compounds with new examples we report here the synthesis of trimethylsilylmethylantimony and on reactions giving a ditellurostibane with the same alkyl group on antimony and p -tolyl groups bound to tellurium. The formation of the

trimethylsilylmethylantimony pentamer as a product of the decomposition of $\text{Me}_3\text{SiCH}_2\text{SbH}_2$ has recently been observed by ^1H NMR spectroscopy [5].

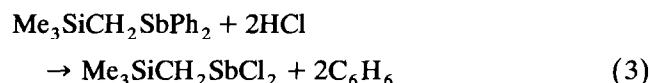
2. Results and discussion

The preparation of trimethylsilylmethylantimony dichloride followed a two-step route [6–8]. The first step implied the formation of diphenyl(trimethylsilylmethyl)antimony (**1**) by reacting a solution of $\text{Me}_3\text{SiCH}_2\text{MgCl}$ in diethylether with Ph_2SbCl [9]:



1

In the second step the phenyl groups in **1** are substituted by chlorine atoms when **1** is reacted with HCl in CHCl_3 :



2

^{*} Dedicated to Professor Herbert Schumann on the occasion of his 60th birthday.

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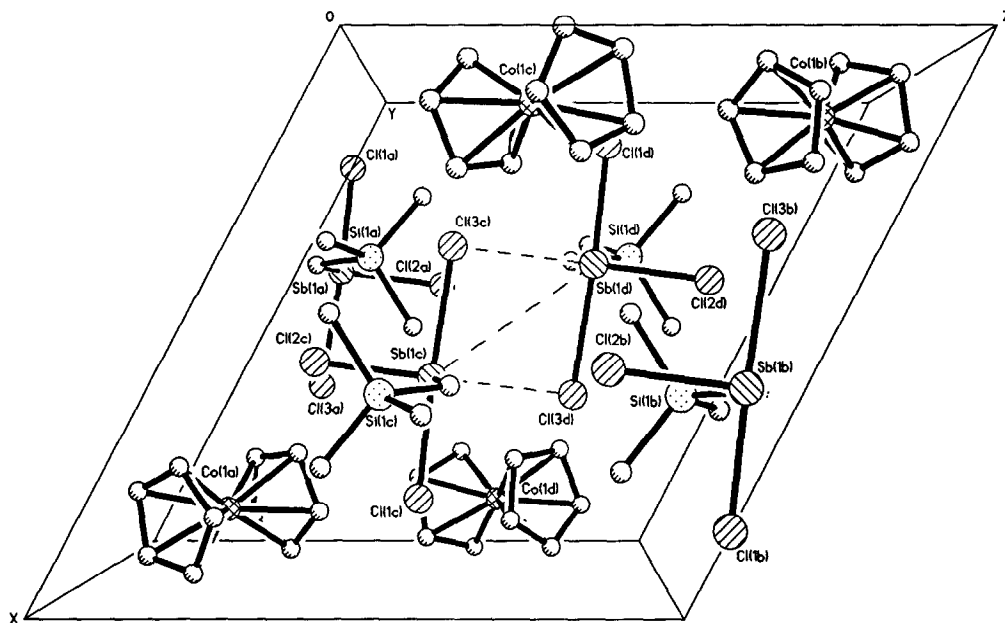
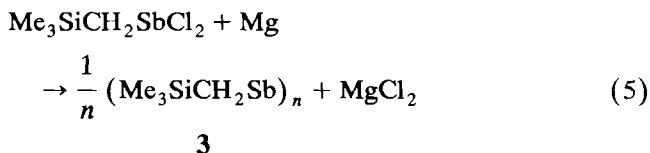


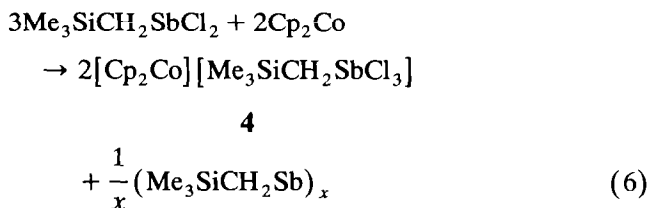
Fig. 1. Unit cell of 4.

After removing the solvent, **2** has been obtained as a yellowish oil, very sensitive to air and moisture. Reduction with magnesium in tetrahydrofuran (THF) resulted in the formation of a black solid and a red–brown oil. After washing this mixture with ethanol from a yellow–orange solution at $-23\text{ }^{\circ}\text{C}$, red crystals of trimethylsilylmethylantimony have been isolated with a 27% yield:



The black solid, isolated with a 60% yield, was characterized as $(\text{Me}_3\text{SiCH}_2\text{Sb})_x$ polymer.

An elegant synthetic approach for the formation of Sb–Sb bonds is the reduction of organoantimony halides with cobaltocene [10]. Stirring a mixture of **2** and Cp_2Co in a 3:2 molar ratio led to a brown solution containing the complex salt $[\text{CoCp}_2][\text{Me}_3\text{SiCH}_2\text{SbCl}_3]$ (**4**) and black solid $(\text{Me}_3\text{SiCH}_2\text{Sb})_x$. No cyclic compound could be identified when the reaction was carried out in THF or in C_6D_6 in an NMR tube:



After removing the solvent, brown–yellow crystals of **4** were isolated with a 87% yield. Recrystallization from ethanol gave crystals suitable for X-ray investigation.

The crystal structure of **4** consists of $[\text{Cp}_2\text{Co}]^+$ cations and $[(\text{Me}_3\text{SiCH}_2\text{SbCl}_3)_2]^{2-}$ anions. The packing of the ions is shown in Fig. 1. The closest interionic distance between Sb and Co is 578.2 pm. The structure of the centrosymmetric anion is shown in Fig. 2. Important distances and angles are listed in Table 1. Table 2 contains the atomic coordinates and the equivalent isotropic displacement coefficients. The anions consist of two trigonal pyramidal RSbCl_2 units bridged by two chloride ions. The Sb–Cl bond lengths lie between 246 and 298 pm. They are longer than the sum of covalent radii ($\sum r_{\text{cov. Sb, Cl}} = 242\text{ pm}$ [11]) but considerably shorter than the sum of the van der Waals contact distances ($\sum r_{\text{v.d.w. Sb, Cl}} = 390\text{--}410\text{ pm}$ [11]). The geometry around the Sb atoms is distorted tetragonal pyramidal with the methylene carbon atoms in apical positions *trans* to the Sb_2Cl_6 planes. When the lone pairs of electrons are considered, the coordination around the antimony atoms

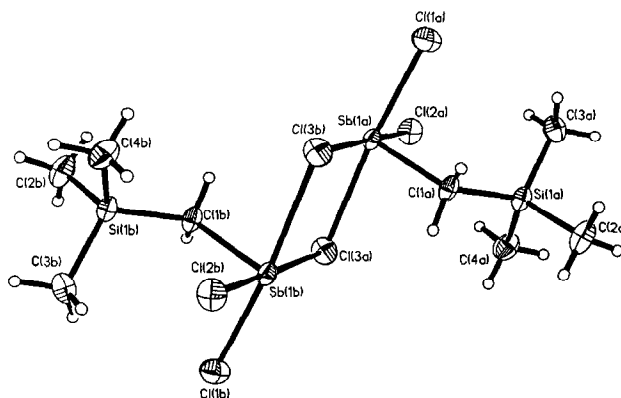


Fig. 2. The structure of the dimers of the anion of 4.

becomes pseudo-octahedral. The antimony atoms lie about 8 pm above the best plane through the neighbouring four Cl atoms as expected for lone-pair repulsion. It should, however, be taken into account that the chlorines atoms themselves deviate pairwise by about 7–8 pm above (Cl(2a) and Cl(3b)) and below (Cl(1a) and Cl(3a)) that plane. Similar geometries around antimony have been observed in the case of various salts containing the $[(\text{PhSbCl}_3)_2]^{2-}$ anion [12,13]. The distance between Cl(2) and Si of 370.2 pm is a little shorter than the sum of van der Waals separation ($\Sigma r_{\text{v.d.w. Si, Cl}} = 380\text{--}400$ [11]) and indicates a very weak interaction. This interaction appears to be compulsory because the angle Sb–C(1)–Si (i.e. 123.4°) is very large and indicates considerable distortion of the tetrahedral carbon atom. Also the angle C–Sb–Cl(2) is relatively large. The structure of the cation does not show unusual features compared with other cobaltocenium salts [10].

The reaction between the cyclostibane **3** and the ditelluride (*p*-TolTe)₂ in THF occurred as expected

Table 1
Important bond distances (pm) and angles (°) in $[\text{Cp}_2\text{Co}][\text{Me}_3\text{SiCH}_2\text{SbCl}_3]$ (**4**)

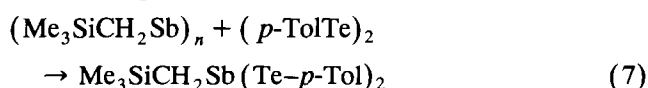
Bond distances			
Sb(1)–C(1)	214.1(4)	Sb(1)–Cl(2)	246.7(1)
Sb(1)–Cl(1)	252.1(1)	Sb(1)–Cl(3a)	280.9(1)
Sb(1)–Cl(3b)	297.7(1)	C(1)–Si(1)	187.2(4)
Si(1)–C(3)	185.8(4)	Si(1)–C(4)	186.4(4)
Si(1)–C(2)	186.8(4)	Co(1)–C(24)	199.1(5)
Co(1)–C(23)	199.2(5)	Co(1)–C(25)	200.9(5)
Co(1)–C(11)	201.7(4)	Co(1)–C(22)	201.7(5)
Co(1)–C(15)	201.8(4)	Co(1)–C(12)	202.7(4)
Co(1)–C(13)	203.3(4)	Co(1)–C(14)	203.7(4)
Co(1)–C(21)	203.7(5)	C(11)–C(15)	138.8(7)
C(11)–C(12)	141.8(7)	C(12)–C(13)	140.4(7)
C(13)–C(14)	139.9(7)	C(14)–C(15)	141.5(7)
C(21)–C(25)	135.1(8)	C(21)–C(22)	137.2(9)
C(22)–C(23)	142.5(10)	C(23)–C(24)	137.3(11)
C(24)–C(25)	135.5(9)	Centre Cp(1)–Co(1)	163.6(4)
Centre Cp(2)–Co(1)	163.4(5)		
Bond angles			
C(1)–Sb(1)–Cl(2)	97.52(10)	C(1)–Sb(1)–Cl(1)	88.04(12)
Cl(2)–Sb(1)–Cl(1)	91.33(5)	C(1)–Sb(1)–Cl(3a)	85.05(11)
Cl(2)–Sb(1)–Cl(3a)	90.91(5)	Cl(1)–Sb(1)–Cl(3a)	172.97(4)
Cl(1)–Sb(1)–Cl(3b)	89.32(5)	Cl(2)–Sb(1)–Cl(3b)	179.25(3)
Cl(3a)–Sb(1)–Cl(3b)	88.40(4)	C(1)–Sb(1)–Cl(3b)	82.1(1)
Si(1)–C(1)–Sb(1)	123.4(2)	Si(1)–C(1)–H(1A)	106.48(13)
Sb(1)–C(1)–H(1A)	106.48(11)	Si(1)–C(1)–H(1B)	106.48(13)
Sb(1)–C(1)–H(1B)	106.48(11)	H(1A)–C(1)–H(1B)	106.5
C(3)–Si(1)–C(4)	109.4(2)	C(3)–Si(1)–C(2)	107.5(2)
C(4)–Si(1)–C(2)	108.8(2)	C(3)–Si(1)–C(1)	113.1(2)
C(4)–Si(1)–C(1)	111.4(2)	C2–Si(1)–C(1)	106.4(2)
C(15)–C(11)–C(12)	107.8(4)	C(13)–C(12)–C(11)	107.6(4)
C(14)–C(13)–C(12)	108.5(4)	C(13)–C(14)–C(15)	107.5(4)
C(11)–C(15)–C(14)	108.6(4)	C(25)–C(21)–C(22)	108.6(5)
C(21)–C(22)–C(23)	107.0(6)	C(24)–C(23)–C(22)	106.1(5)
C(25)–C(24)–C(23)	109.1(6)	C(21)–C(25)–C(24)	109.2(6)
Centre Cp(1)–Co(1)	177.3(3)		
–Centre Cp(2)			

Table 2
Atom coordinates and equivalent isotropic displacement coefficients

	<i>x</i> ($\times 10^4$)	<i>y</i> ($\times 10^4$)	<i>z</i> ($\times 10^4$)	U_{eq}^a ($\times 10^4$ pm ²)
Sb(1)	3864(1)	41(1)	909(1)	21(1)
Cl(1)	1485(1)	38(1)	14(1)	37(1)
Cl(2)	4176(1)	–77(1)	3146(1)	32(1)
Cl(3)	6481(1)	–170(1)	1780(1)	33(1)
C(1)	3753(4)	–1367(2)	498(4)	24(1)
Si(1)	3631(1)	–2247(1)	1580(1)	23(1)
C(2)	3329(5)	–3332(3)	683(4)	38(1)
C(3)	2289(4)	–2053(3)	1943(4)	34(1)
C(4)	5136(4)	–2343(3)	3127(4)	36(1)
Co(1)	8879(1)	6505(1)	1538(1)	22(1)
C(11)	8902(6)	5152(3)	1494(5)	44(1)
C(12)	10063(4)	5500(3)	1621(4)	40(1)
C(13)	9776(5)	6047(3)	539(5)	37(1)
C(14)	8461(5)	6036(3)	–256(4)	38(1)
C(15)	7928(5)	5481(3)	348(5)	41(1)
C(21)	9193(6)	7838(3)	1953(5)	48(1)
C(22)	7922(6)	7647(4)	1421(6)	70(2)
C(23)	7773(8)	7001(5)	2241(10)	93(3)
C(24)	8970(10)	6841(4)	3229(7)	76(2)
C(25)	9818(5)	7340(4)	3036(5)	52(1)

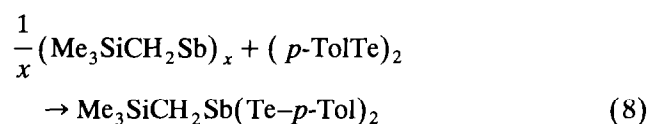
^a Equivalent isotropic U_{eq} defined as one third of the trace of the orthogonalized U_{ij} tensor.

with formation of the ditellurostibane $\text{Me}_3\text{SiCH}_2\text{Sb}(\text{Te}-p\text{-Tol})_2$ (**5**) with a quantitative yield:

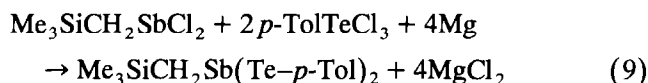


5

The same tellurostibane has also been obtained either by reacting the black polymer $(\text{Me}_3\text{SiCH}_2\text{Sb})_x$ with $(p\text{-TolTe})_2$ according to



or by reducing a mixture of RSbCl_2 and $p\text{-TolTeCl}_3$ in a 1:2 molar ratio in THF with magnesium



Both reactions gave **5** only with moderate or low yields as mixtures with $(p\text{-TolTe})_2$ or **3**. The formation of **5** in reaction (8) is, however, significant as proof of the presence of $(\text{Me}_3\text{SiCH}_2\text{Sb})_x$ in the black solids formed in Eqs. (5) and (6).

The identity of **1–5** is proven by elemental analyses and spectroscopic investigations. The ¹H NMR spectra of **3** in C₆D₆ solution are characteristic for the presence of an equilibrium mixture of the five- and four-membered rings. The five-membered ring $(\text{Me}_3\text{SiCH}_2\text{Sb})_5$ is characterized by three singlet signals corresponding to the trimethylsilyl groups in a 2:2:1 ratio of intensities,

by the signals of two AB spin systems corresponding to two pairs of diastereotopic methylene groups, and by a singlet signal for the fifth methylene group. This pattern is characteristic for an antimony five-ring with a time-equalized plane of symmetry with organo substituents adopting a maximum of *trans* positions. Similar results have been reported by Wells et al. [14] for $(\text{Me}_3\text{SiCH}_2\text{As})_5$ and by Hendershot and Berry [5] for $(\text{Me}_3\text{SiCH}_2\text{Sb})_5$. In addition to the signals of the five-ring there are two singlet signals in a 9:2 ratio of intensities. We attribute these signals to the novel four-membered ring $(\text{Me}_3\text{SiCH}_2\text{Sb})_4$ because their relative intensity is increasing on dilution as expected for equilibria between five- and four-rings (Eq. (1)) following the principle of Le Chatelier. The mass spectrum of **3** contains signals not only of pentamers and tetramers but also of trimers and of rearrangement products and thus does not allow the determination of the ring size in condensed phases. The ring size in the crystalline state is yet unknown. The crystals of **3** were not suitable for X-ray analyses. From the colour and solubility it can, however, be excluded that solid **3** is a polymer like all the other alkyl antimony ring systems consisting of a pentamers and tetramers in solution. Another property of **3** that is remarkable is the colour change between the crystalline state (red) and the solution (yellow). Similar changes in colour have been observed in the case of distibanes [2,15] and a cyclotristibane [16]. The novel tellurostibane **5** does not exhibit unusual colour changes.

3. Experimental details

All the experiments have been done in an argon atmosphere, using dried solvents distilled under argon. The ^1H NMR spectra were recorded in C_6D_6 on a Bruker WH 360 instrument operating at 360 MHz. The electron impact (EI) mass spectroscopy (MS) was carried out on a Varian MAT CH7 (A) and the fast atom bombardment (FAB) MS on a Finnigan MAT 8222 instrument. Elemental analyses were performed by Mikroanalytisches Laboratorium Beller in Göttingen.

3.1. Preparation of diphenyl(trimethylsilylmethyl)antimony (**1**), trimethylsilylmethylantimony dichloride (**2**) and trimethylsilylmethylantimony (**3**)

A solution of 30.0 g (96 mmol) of Ph_2SbCl in 100 ml of diethyl ether was added dropwise to a Grignard solution prepared from 11.8 g (96 mmol) of $\text{Me}_3\text{SiCH}_2\text{Cl}$ and 2.5 g (0.1 mol) of magnesium in 100 ml of diethyl ether. After stirring for 16 h at room temperature the reaction mixture was hydrolysed with 150 ml of water saturated with argon. The organic phase was separated and the water phase was washed twice with 100 ml of diethyl ether. The organic phases were dried with Na_2SO_4 . Removal of the solvent under

reduced pressure gave 31.7 g (90.6%) of **1** as a yellowish oil. ^1H NMR (C_6D_6): δ 0.06 (s, 9H, Me_3Si), 0.90 (s, 2H, CH_2), 7.1–7.2, 7.5–7.6 (m, 10H, C_6H_5) ppm. MS (EI, 70 eV, 70°C): m/z (%) 362 (32) M^+ , 285 (57) $\text{M}^+ - \text{Ph}$, 275(19) Ph_2Sb , 208 (21) $\text{Me}_3\text{SiCH}_2\text{Sb}$, 135 (100) Me_2SiPh . A stream of gaseous hydrogen chloride was bubbled for 3 h through a solution of 31.7 g (87 mmol) of **1** in 150 ml of chloroform. After all the unreacted HCl had been removed from the flask, the solvent was distilled under reduced pressure and 23.9 g (98%) of **2** remained as a yellowish oil. **2**: ^1H NMR (C_6D_6): δ 0.01 (s, 9H, Me_3Si), 1.05 (s, 2H, $-\text{CH}_2-$) ppm. MS (EI, 70 eV, 20°C): m/z (%) 265 (100) $\text{M}^+ - \text{Me}$, 245 (6) $\text{M}^+ - \text{Cl}$, 208 (7) $\text{Me}_3\text{SiCH}_2\text{Sb}$, 193 (8) $\text{Me}_2\text{SiCH}_2\text{Sb}$, 165 (17) SiCH_2Sb , 156 (4) SbCl , 135 (23) SbCH_2 , 73 (66) Me_3Si .

A solution of 16.0 g (0.057 mol) of **2** in 70 ml of THF was added dropwise to 1.4 g (0.057 mol) of magnesium filings. The reaction mixture was stirred for 5 h until all the magnesium had reacted. The solvent was removed in vacuo and the black remaining product mixture was washed with petroleum ether, giving an orange-brown solution. The black solid residue was washed with water, filtered and washed again with diethyl ether; and then it was dried under reduced pressure. 7.4 g of a black powder was obtained. The solvent was removed from the solution petroleum ether and a brown oil was obtained. Addition of ethanol gave a yellow solution. Cooling for 16 h at -23°C gave 3.2 g (26.8%) of **3** as red crystals (melting point, $78-80^\circ\text{C}$). ^1H NMR (C_6D_6): δ ($\text{Me}_3\text{SiCH}_2\text{Sb})_5$: 0.17 (s, 18H, CH_3), 0.18 (s, 9H, CH_3), 0.19 (s, 18H, CH_3), AB spin system with A: 1.52, B: 1.68 ($^2J = 13$ Hz, 4H, $-\text{CH}_2-$), AB spin system with A: 1.40, B: 1.57 ($^2J = 13$ Hz, 4H, $-\text{CH}_2-$) 1.47 (s, 2H, $-\text{CH}_2-$); ($\text{Me}_3\text{SiCH}_2\text{Sb})_4$: 0.28 (s, 36H, Me_3Si), 1.39 (s, 8H, $-\text{CH}_2-$) ppm. MS (EI, 70 eV, 220°C): m/z (%) 957 (2) R_4Sb_5 , 834 (2) R_4Sb_4 , 749 (5) R_3Sb_4 , 626 (30) R_3Sb_3 , 539 (14) R_2Sb_3 , 296 (54) R_2Sb , 207 (30) RSb , 73 (100) Me_3Si . Anal. Found: C, 23.44; H, 4.85; $\text{C}_4\text{H}_{11}\text{SbSi}$ calc.: C, 23.07; H, 5.29%.

3.2. Preparation of bis(cyclopentadienyl)cobalt trichloro(trimethylsilylmethyl)antimonate (**4**)

A mixture of 0.5 g (1.8 mmol) of **2** and 0.22 g (1.2 mmol) of Cp_2Co in THF was stirred for 1 h at room temperature. A black solid precipitated and the solution became yellow-brown. The solution was separated from the solid and the solvent was removed. Recrystallization from ethanol gave 0.52 g (86.6%) of **4** as yellow-brown crystals. ^1H NMR (C_6D_6): δ 0.12 (s, 9H, Me_3Si), 1.3 (s, 2H, $-\text{CH}_2-$), 5.86 (s, 10H, C_5H_5) ppm. MS (FAB): m/z (%) 189 (73) Cp_2Co , 315 (54) $\text{Me}_3\text{SiCH}_2\text{SbCl}_3$, 73 (100) Me_3Si . Anal. Found: C, 33.25; H, 4.16, $\text{C}_{14}\text{H}_{21}\text{Cl}_3\text{CoSbSi}$ calc.: C, 33.36; H, 4.17%.

3.3. Preparation of trimethylsilylmethylbis(*p*-tolyltelluro)antimony (5)

(a) A mixture of 0.21 g (0.48 mol) of (*p*-TolTe)₂ and 0.1 g (0.48 mol) of **3** in THF was stirred at room temperature for 1 h. Removal of the solvent and crystallization from the orange solution in ethanol gave 0.31 g (100%) of **5** as an orange powder. ¹H NMR (C₆D₆): δ 0.29 (s, 9H, Me₃Si), 1.43 (s, 2H, –CH₂–), 1.98 (s, 6H, *p*-CH₃), 6.70–6.75, 7.78–7.81 (AA'BB' spin system, 8H, C₆H₄) ppm. MS (EI, 70 eV, 170°C): *m/z*(%) 648 (19) M⁺, 427 (40) Me₃SiCH₂SbTeC₇H₇, 221 (30) C₇H₇Te, 91 (100) C₇H₇. Anal. Found: C, 33.34; H, 3.97. C₁₈H₂₅SiSbTe₂, calc.: C, 33.44; H, 3.87%.

(b) A mixture of **2** and *p*-TolTeCl₃ in a 1:2 molar ratio has been reduced with magnesium in a THF solution by stirring for 12 h. The THF was removed under reduced pressure and the black residue was washed with petroleum ether. After removing the solvent from the orange solution an orange oil was obtained. It was spectroscopically characterized as a mixture of **3**, **5**, and (*p*-TolTe)₂.

(c) Stoichiometric amounts of the black polymer (RSb)_x and (*p*-TolTe)₂ were stirred in THF for 16 h at room temperature. An orange solution was separated from the great quantity of unreacted polymer. Work-up as described above gave a mixture of **5** and (*p*-TolTe)₂.

3.4. Crystal structure determination

3.4.1. Crystal data

C₁₄H₂₁Cl₃CoSbSi; *M* = 504.43; monoclinic; *a* = 1191.2(2) pm, *b* = 1489.6(3) pm and *c* = 1167.9(2) pm; β = 117.84(3)°; *V* = 1.8325(6) nm³, *Z* = 4, *D*_c = 1.828 g cm⁻³; μ = 2.870 mm⁻¹; *F*(000) = 992; space group, *P*2₁/*c*; crystal size 0.80 × 0.60 × 0.50 nm³.

3.4.2. Structure determination

Data were collected on a Siemens P4 four-circle diffractometer at 173(2) K with Mo Kα radiation, λ = 71.073 pm for 5055 reflections in the 2θ–ω mode, of which 4020 were independent reflections (*R*_{int} = 1.86%) and 4004 were used in the full-matrix least-squares refinement with the SHELXL-93 [17] program system. The structure was solved by direct methods for antimony and the difference Fourier synthesis revealed the positions for the other non-hydrogen atoms. All non-hydrogen atoms were refined anisotropically and hydrogen atoms were calculated as a riding model with isotropic *U* thermal parameters. The final *R* values are *R*₁ = 3.96% and *wR*₂ = 9.76% (*R*₁ = 5.004% and *wR*₂ = 10.72% for all data), with goodness of fit 0.955. The

residual electron density from a final difference Fourier synthesis was in the range from –2000 to 1426 electrons nm⁻³. Refined values of the atomic coordinates are given in Table 2. Further details of the crystal structure investigation can be obtained from the Fachinformationszentrum Karlsruhe, Gesellschaft für wissenschaftlich technische Information mbH, D-76344 Eggenstein-Leopoldshafen, Germany, on quoting the deposit number CSD 58925, the names of the authors and the journal citation.

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