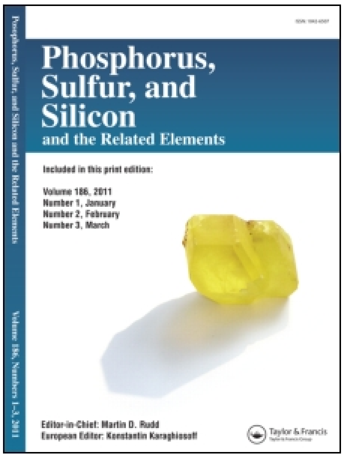

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Complexes of Cyclostibane-Ligands

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The reaction of *cyclo*-$(Me_3SiCH_2Sb)_5$ with excess of $W(CO)_5$thf (thf = tetrahydrofuran), in thf gives the 1:2 complex: *cyclo*-$(Me_3SiCH_2Sb)_5$ 1,3-$[W(CO)_5]_2$. A X-ray crystal structure analysis reveals that the complex contains a five membered antimony ring in the envelope conformation. The $W(CO)_5$ fragments are in trans positions.

Keywords: antimony ligands; organoantimony compounds; tungsten complexes

INTRODUCTION

Organoantimony monocycles $(RSb)_n$ [1] are known for n = 3, 4, 5, 6 (R = alkyl, aryl). Rings with n = 3 [2], 4 (R = $(Me_3Si)_2CH$, ^tBu, Mes), n = 6 (R = Ph, tolyl) have been characterized by X-ray diffraction. Attempts to determine the crystal structure of $(Me_3SiCH_2Sb)_5$ failed. $(RSb)_5$ (R = Et, Pr, Bu, Ph, tolyl) exist only in equilibra in solution. Sb-5-rings are known in the polycycle R_4Sb_8 (R = $(Me_3Si)_2CH$) [3].

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Known complexes with cyclostibane ligands are $^{t}Bu_4Sb_4M(CO)_5$ (M = Mo, W), $^{t}Bu_4Sb_4$-1,3-$[M(CO)_5]_2$, $^{t}Bu_4Sb_4Fe(CO)_4$ [4].

We report here the synthesis and structure of the first complex with the *cyclo*-$(Me_3SiCH_2)_5Sb_5$ ligand and the first crystal structure of a monocyclic R_5Sb_5 derivative.

RESULTS AND DISCUSSION

cyclo-$(Me_3SiCH_2)_5Sb_5$ was prepared according to ref. [5]. The reaction of *cyclo*-$(Me_3SiCH_2)_5Sb_5$ with $W(CO)_5$thf (1:3 molar ratio) in thf (eq. 1) gives the 1:2 complex, $(Me_3SiCH_2Sb)_5[W(CO)_5]_2$ in 70% yield, as yellow air sensitive solid, soluble in common organic solvents. Solutions decompose when heated to 60 °C. Single crystals (m.p. 138 °C) are obtained by recrystalisation from petroleum ether.

$$(RSb)_5 \xrightarrow[-\,2\ \mathrm{thf}]{+\,2\ \mathrm{thfW(CO)_5}} (RSb)_5\text{-1,3-}[W(CO)]_2 \quad (1)$$

$$R = Me_3SiCH_2$$

A X-ray structure analysis was carried at – 80 °C out to determine the structure of the complex. The molecular structure of

(Me$_3$SiCH$_2$Sb)$_5$–1,3-[W(CO)$_5$]$_2$ in the crystal is depicted in Fig.1. The complex consists of a five membered antimony ring in the envelope conformation. The cyclostibane ligand is coordinated to two pentacarbonyltungsten units in the 1,3 positions. The alkyl substituents of the cyclostibane adopt a maximum of trans positions. The average of Sb-Sb- bond length is 2.825 Å and the average of the Sb-W distances is 2.803 Å. Both values compare very well with the Sb-Sb-, and Sb-W distances respectively in ^{t}Bu$_4$Sb$_4$-1,3-[W(CO)$_5$]$_2$ [4].

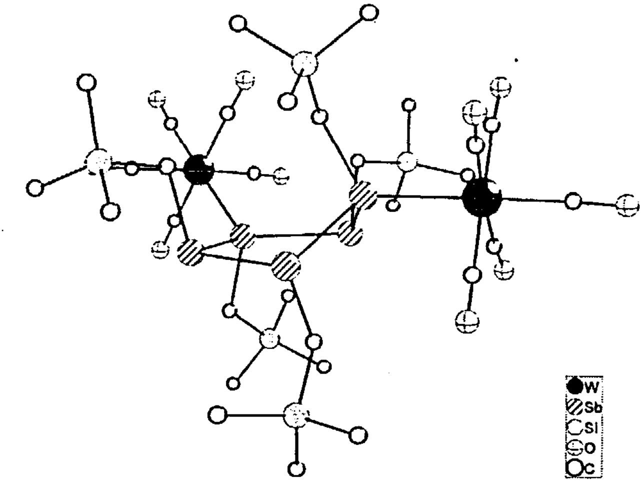

Fig. 1 Crystal structure of *cyclo*-(Me$_3$SiCH$_2$Sb)$_5$–1,3-[W(CO)$_5$]$_2$. The hydrogen atoms are omitted for clarity.

In the ^1H NMR spectrum of $(Me_3SiCH_2Sb)_5[W(CO)_5]_2$ there are five singlet signals of equal intensities for the methyl groups and ten doublet signals for the diastereotopic methylene groups. In the IR spectrum in benzene there are two signals at 2064 and at 1942 cm^{-1} for the νCO vibrations of the $W(CO)_5$ units.

Acknowledgements

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