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New stibines containing acetal and formyl group: Platinum complex and unexpected oxastibol derivative

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Abstract

A new tertiary stibine ligand (**1**) containing acetal group at the *ortho*-position has been synthesized. This new stibine was then complexed with $PtCl_4^{2-}$ to obtain *trans*-$PtCl_2L_2$ (**2**) where stibine acts as a monodentate ligand. The acetal was hydrolyzed in a slightly acidic medium and forms a very new stibine (**3**) containing formyl group at the *ortho*-position. When (**3**) was reduced with $NaBH_4$ an unusual oxastibol (**4**) derivative was obtained under the experimental conditions used.

All the compounds were characterized by IR, mass, ^1H, ^{13}C, COSY and HETCOR NMR spectroscopy. The molecular structures of (**2**), (**3**) and (**4**) were determined. Compound (**3**) crystallizes in two different polymorphs (**3a**) and (**3b**) and both the polymorphs show hypervalent antimony with three Sb···O interactions and the molecule exists in *O-cis-exo* configuration. Compound (**4**) shows intramolecular Sb···O interactions. To the best of our knowledge this is the first report on organoantimony compounds containing carbonyl groups though their phosphorus and bismuth analogues are well known. Compound (**2**) shows helicoidal chirality, which is a very new concept in antimony chemistry.

Keywords: Hypervalent; Stibines; Intramolecular Sb–O interactions; Chiral molecule; Pt–Sb complexes

1. Introduction

Slight shortening of the inter atomic distances in comparison to the sum of the van der Waals radii of group 14–16 elements and other hetero atoms such as oxygen or nitrogen have found in certain compounds. This hypervalent interaction is of interest because of the effect it may have on the structures, chemical properties and biological activities [1–8]. Most of the interactions involving group 15 elements were observed in compounds with a cyclic structure, suggesting the influence of steric factors [9–11]. Recently our group has reported some new stibines containing CH_2NMe_2 pendant arm in the *ortho*-position, which also show these interactions [12].

Because of our interest in functionalized triarylstibines ligands for their applications in catalysis with rhodium, cobalt and ruthenium, especially those comprising triple substitution in *ortho*-positions [13–15], here we wish to report the preparation, structural characterization of some Sb (III) compounds containing Sb···O interactions this work was undertaken.

2. Results and discussion

The colorless crystalline stibine **1** was prepared via a salt elimination reaction of *o*-lithiated benzaldehyde diethylacetal with $SbCl_3$ in THF at −78 °C. Stibine **1** forms complex $PtCl_2 \cdot L_2$ (**2**) on reaction with $[PtCl_4]^{2-}$. Acidic hydrolysis of (**1**) produces yellow colored tris(2-formylphenyl)stibine (**3**). When stibine (**3**) was reduced with $NaBH_4$ in ethanol water mixture, an unexpected oxastibol (**4**) was obtained (Scheme 1). In order to isolate tris[(2-hydroxymethyl)-

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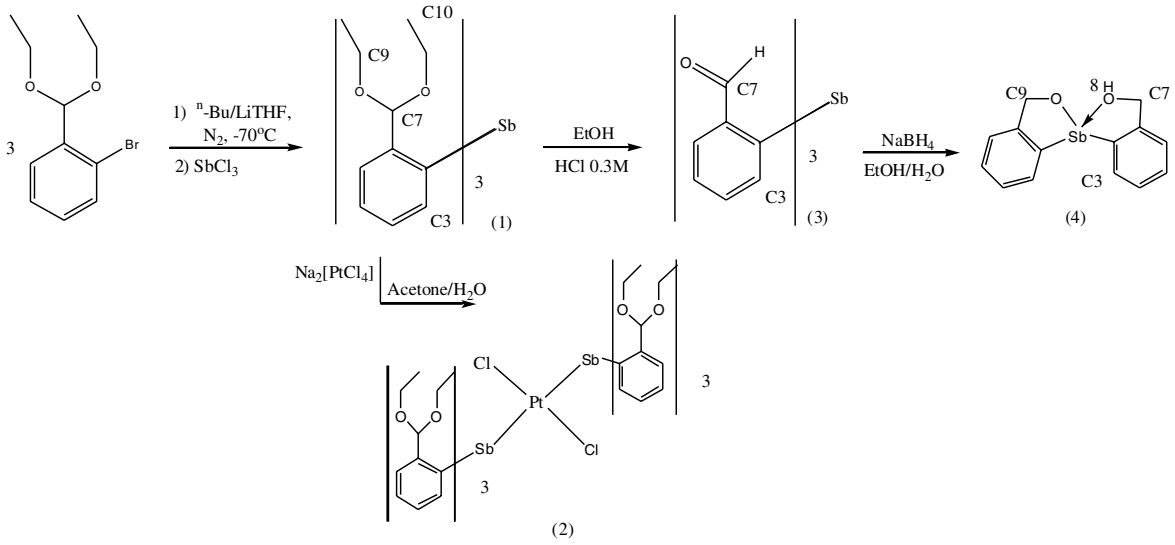

Scheme 1.

phenyl]stibine, the reduction was carried out for 30 min, 2 h and 4 h. Under the experimental conditions used, only the yield of the oxastibol product (**4**) changes marginally with the time of the reaction and attempts to obtain the tris[(2-hydroxymethyl)phenyl]stibine failed. It is to be noted that under the similar reaction conditions tris[(2-hydroxymethyl)phenyl]phosphine [16] was obtained from the corresponding formylphosphine.

All the compounds are stable and melts without decomposition and are soluble in polar organic solvents, e.g., chloroform , dichloromethane, ether and are insoluble in water and non polar solvents, e.g., hexane, pentane It is important to mention that Sb–C bonds in these stibines do not break in slightly acidic aqueous medium, but in general, Sb–C bond is sensitive to such conditions and breaks.

Molecular ion peaks were observed for all compounds in the FAB^+ MS, along with significant fragments arising from Sb–C cleavage. In all the cases the assignment of individual protonic signals in the ^1H NMR spectra was based on J_{HH} coupling constant values and was confirmed by COSY and HETCOR. Though Sb···O interactions were observed in the structural characterizations, evidence for such interactions could not be detected in ^1H NMR spectra of compound (**3**). For the compound (**2**), ^1H NMR signals appeared at slightly downfield in comparison to the free ligand. Compounds (**1**) and (**3**) show higher chemical shift of aromatic protons [(7.79 ppm for compound (**1**) and 7.53 ppm for compound (**3**)] in comparison to the corresponding phosphorus compounds [7.6 ppm for tris(2-formylphenyl)phosphine and 6.87 ppm for tris(1,3-dioxaolan-2-yl phenyl)phosphine) [17,18]. Similar to ^1H NMR , in ^{13}C NMR a higher chemical shift were observed in stibines in comparison to reported phosphines. For compound (**4**), a chemical shift at 4.61 ppm corresponds to methylene protons (H7) which is at slightly downfield in comparison to the chemical shift (4.58 ppm) observed in a similar compound of phosphorus namely tris [2-(hydroxymethyl)phenyl]phosphine. Similarly H3 protons show a more downfield shift in antimony compound (7.91 ppm) than in phosphorus (7.62 ppm). A coupling between H9 protons with the hydroxyl proton (H8) was also observed, which may be due to Sb···O intramolecular interaction (see Fig. 1).

The molecular structures of **2**–**4** have been confirmed by X-ray crystallography. Tables 1 and 2 summarize the crystal data and bond lengths and bond angles for these compounds, respectively. All these compounds are monomeric in nature and no significant intermolecular interactions were observed, except compound (**4**). Compound (**3**) crystallizes in two different crystalline forms: trigonal form with $P\bar{3}$ space group (**3a**) with chloroform as a solvent of crystallization and rhombohedral form with *R3* space group (**3b**) with toluene as a solvent of crystallization. In both the polymorphs, molecule is chiral and compound crystallizes with two independent molecules per asymmetric unit (Figs. 2 and 3). The metrical parameters and stereochemical features of the two independent molecules are very similar. It is to be mentioned here that very few examples are known in organoantimony compounds showing polymorphism. In both the polymorphs, three Sb···O intramolecular interactions were observed and the three formyl groups adopt *O-cis-exo* conformation. It is to be noted that in the phosphorus analog, two of the three formyl groups possess the *O-cis-exo* while the third formyl group was best described as *O-trans-exo*. This may be due to the Sb···O intramolecular interactions observed in (**2**) in lieu hypervalency of antimony.

The primary coordination sphere consists of a trigonal pyramidal SbC_3 skeleton, and including the three Sb···O interactions the antimony can be described as heptacoordinate with one position occupied by a lone pair of electrons. It is to be noted that heptacoordination in antimony is not very common.

In the compound (**4**) , the distance between oxygen atom of O–H group and the central antimony atom is 2.458(3) Å, which is much shorter than the sum of their van der Waals

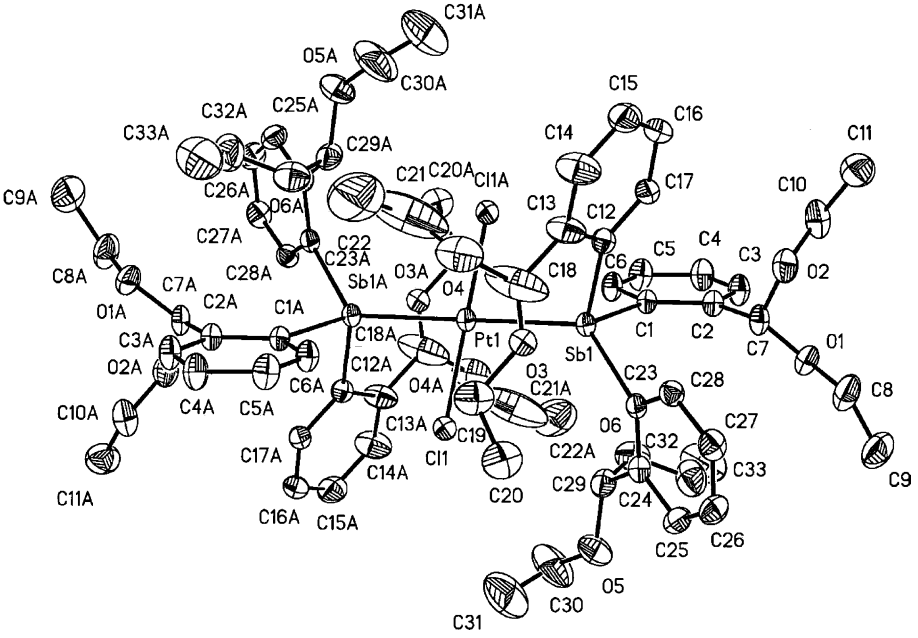

Fig. 1. Molecular structure of compound (**2**).

Table 1
Crystal data for compounds (**2**), (**3a**), (**3b**) and (**4**)

Compounds	(**2**)	(**3a**)	(**3b**)	(**4**)
Empirical formula	$C_{66}H_{90}Cl_2O_{12}PtSb_2$	$C_{21}H_{15}O_3Sb,CHCl_3$	$C_{21}H_{15}O_3Sb,0.3,C_7H_8$	$C_{14}H_{13}O_2Sb$
Formula weight	1584.87	556.45	467.12	334.99
Crystal system	Triclinic	Trigonal	Rhombohedral	Orthorhombic
Space group	$P\bar{1}$	$P\bar{3}$	$R3$	$Pbca$
Crystal size	$0.212 \times 0.196 \times 0.164$	0.162×0.158x0.136	$0.318 \times 0.278 \times 0.182$	$0.134 \times 0.066 \times 0.064$
a (Å)	10.6275(6)	12.1779(3)	12.3310(4)	8.6368(6)
b (Å)	12.3149(7)	12.1779(3)	12.3310(4)	12.3974(8)
c (Å)	142174(8)	17.258(1)	26.517(2)	23.253(2)
α (°)	101.503	90	90	90
β (°)	91.730	90	90	90
γ (°)	108.186	120	120	90
V (Å^3)	1723.7(2)	2216.5(2)	3491.8(3)	2489.8(3)
Z	1	4	6	8
D_{calc} (Mg/cm^3)	1.527	1.668	1.333	1.787
μ (mm^{-1})	2.936	1.627	1.202	2.202
Reflections collected	33943	18469	9581	18822
Independent reflections	12248	2650	2744	2195
$R[I > 2(I)]$	0.0527	0.0378	0.0588	0.0354
GOF	0.992	0.981	1.002	0.990
$\Delta/\sigma e$ (Å^{-3})	2.109/−0.831	0.770/−0.571	2.026/−0.453	0.943/−0.502

radii. Thus, the coordination of O–H group forms a hypervalent 10-Sb-4 compound (Fig. 4). The geometry about antimony atom is distorted pseudotrigonal bipyramid where the two carbon atoms bound to antimony atom occupy the equatorial plane. The apical positions are occupied by two oxygen atoms, while the lone pair of electrons can be considered to occupy the equatorial position. There exists intermolecular hydrogen bonding O–H···O [1.72(4) Å] in the crystal structure.

In complex (**2**), the stibine ligand (**1**) acts as a monodentate ligand. Platinum is in square planar geometry, and the two stibine ligands lie in a *trans* configuration (Fig. 1). To best of our knowledge very few X-ray structures of platinum stibine complexes have been reported in the literature. The average Pt–Cl bond length in (**2**) is 2.293(11) Å, which is slightly shorter than the average Pt···Cl distance in *cis*-$[PtCl_2(SbPh_3)_2]$ 2.338(12) Å. The Pt–Sb bond length is slightly [2.582(3)Å] longer than found in *trans*-$[PtBrPh(SbPh_3)_2]$ [2.548(1) Å] and *trans*-$[PtI_2(SbPh_3)_2]$ [2.552(1) Å] [19,20].

3. Experimental

All the solvents were distilled immediately prior to use and the reactions were performed under an atmosphere of oxygen-free, dry nitrogen. Elemental analyses were determined

Table 2
Selected bond lengths (Å) and bond angles (°)

*Compound (**2**)*			
Sb(1)–Pt(1)	2.5821(3)	Sb(1A)–Pt(1)	2.5821(3)
Sb(1)–C(12)	2.151(4)	Sb(1)–C(1)	1.153(4)
Pt(1)–Cl(1A)	2.2925(11)	Pt(1)–Cl(1)	2.2925(11)
Sb(1)–C(23)	1.143(4)	Cl(1A)–Pt(1)–Sb(1)	84.40(3)
Cl(1)–Pt(1)–Cl(1A)	180.0(7)	Cl(1A)–Pt(1)–Sb(1A)	95.60(3)
Cl(1)–Pt(1)–Sb(1)	95.60(3)	Sb(1A)– Pt(1)– Sb(1)	180.0(11)
Cl(1)–Pt(1)–Sb(1A)	84.40(3)	C(12)–Sb(1)–Pt(1)	107.49(11)
C(23)–Sb(1)–Pt(1)	127.66(10)	C(1)– Sb(1)–Pt(1)	110.00
*Compound (**3a**)*			
Sb(1)–C(1)	2.207(16)	Sb(1)–C(1A)	2.207(16)
Sb(1)–C(1B)	2.207(16)	Sb(1)–O(2)	2.899(15)
Sb(1)–O(1)	2.899(13)	Sb(2)–C(8)	2.128(15)
Sb(2)–C(8A)	2.128(16)	Sb(2)–C(8A)	2.128(16)
C(1)–Sb(1)–C(1A)	96.1(6)	C(1)–Sb(1)–O(1)	68.4(5)
C(1)–Sb(1)–C(1B)	96.1(6)	C(1A)–Sb(1)–O(1)	163.4(5)
C(1A)–Sb(1)–C(1B)	96.1(6)	C(1B)–Sb(1)–O(1)	80.4(5)
C(8A)–Sb(2)–O(2)	157.9(5)	C(8A)–Sb(2)–C(8)	93.1(6)
C(8)–Sb(2)–O(2)	67.4(5)	C(8A)–Sb(2)–C(8B)	93.1(6)
C(8B)–Sb(2)–O(2)	78.2(6)	C(8)–Sb(2)–C(8B)	93.1(6)
O(1)–C(7)–C(2)	131.4(15)		
*Compound (**3b**)*			
Sb(1)–C(1)	2.190(4)	Sb(1)–C(1A)	2.190(4)
Sb(1)–C(1B)	2.190(4)	Sb(1)–O(2)	2.883(3)
Sb(2)–O(1)	2.850(3)	Sb(2)–C(8)	2.186(4)
Sb(2)–C(8A)	2.186(4)		
C(1A)–Sb(1)–C(1B)	94.72(13)	C(1)–Sb(1)–O(1)	68.48(12)
C(1A)–Sb(1)–C(1)	94.72(13)	C(1A)–Sb(1)–O(1)	77.80(12)
C(1B)–Sb(1)–C(1)	94.72(13)	C(1B)–Sb(1)–O(1)	160.62(12)
C(8A)–Sb(2)–O(2)	161.79(12)	C(8A)–Sb(2)–C(8)	95.56(13)
C(8)–Sb(2)–O(2)	68.09(12)	C(8A)–Sb(2)– C(8B)	95.56(14)
C(8B)–Sb(2)–O(2)	78.82(12)	C(8B)–Sb(2)– C(8)	95.56(14)
*Compound (**4**)*			
Sb(1)–C(1)	2.128(4)	Sb(1)–C(8)	2.145(4)
Sb(1)–O(1)	2.056(3)	Sb(1)–O(2)	2.458(3)
O(1)–C(7)	1.403(5)	O(2)–C(14)	1.422(5)
C(3)–C(4)	1.367(7)	C(1)–Sb(1)–O(2)	81.46(14)
C(1)–Sb(1)–C(8)	96.56(15)	O(1)–Sb(1)–C(8)	90.97(14)
O(1)–Sb(1)– C(1)	80.45(15)	O(1)–Sb(1)–O(2)	154.60(12)

on a Perkin–Elmer 240 instrument. Melting points were obtained using a MEL-TEMP II Fisher and are uncorrected. EI and FAB^+ mass spectra were recorded on a JEOL SX102 double-focusing mass spectrometer with reverse geometry using a 6-kV Xenon beam (10 am); nitrobenzyl alcohol was used as matrix for recording the mass spectra. IR spectra were recorded on a Nicolet-Magna 750 FT-IR spectrometer as nujol mulls. ^1H (300.5311 MHz) and ^{13}C (75.5757 MHz) NMR spectra were recorded in $CDCl_3$ on a JEOL ECLIPSE 300 spectrometer.

3.1. X-ray crystallography

The X-ray intensity data were measured at 293 °K on a Bruker SMART APEX CCD-based X-ray diffractometer using a monochromatized Mo Kα radiation (Kα = 0.71073 Å). The detector was placed at a distance of 4.837 cm from the crystal in all the cases. Analyses of data show negligible decay during the data collections. An analytical face indexed absorption correction was applied. Crystal structures were refined by full-matrix least-squares. SMART software (data collection and data reduction) and SHELXTL program were used for solution and refinement of the structures.

3.2. Syntheses

3.2.1. Tris(2-diethylacetal formyl phenyl)stibine (**1**)

In a Schlenk tube a solution of antimony trichloride (0.485 g, 2.13 mmol) in anhydrous THF (10 ml) was added dropwise under a nitrogen atmosphere to 2-lithiobenzaldehyde diethylacetal which was synthesized "in situ" according to reported method (to 4 ml of ^{n}BuLi (2.5 M in hexane) under inert atmosphere, a solution of 1.89 ml (9 mmol) of 2-bromobenzaldehyde diethyl acetal in 10 ml of THF at −78 °C was added dropwise for 1 h in THF −20 °C with continuous stirring). The mixture was further stirred for 2 h at room temperature and then reaction was quenched with ice. After extraction with dichloromethane (3 × 10 ml) and drying over sodium sulfate, solvent was removed under vacuum. Slow concentration from chloroform solution obtains colorless microcrystalline compound. Yield: 0.92 g (80%); m.p.: 96–97 °C; Anal. Found (Calc.): C, 59.69 (60.18); H, 6.26 (6.83)%. For $C_{33}H_{30}O_6Sb$; IR (ν cm^{-1}): 476 (Sb–C), 2979 (C–H aromatic), 1058, 1096 (C–O–C); FAB^+ m/z 658 (3%) $[M]^+$, 479(12%) $[M-L]^+$, 358 (34%) $[L-L]^+$, 302 (5%) $[M-2L]^+$, 179 (23%) $[L]^+$; NMR ^1H ($CDCl_3$, δ in ppm) 7.7853 (3H, d, H3J_{HH} 7.5 Hz), 7.2943 (6H, m, H4, H6), 7.0590 (3H, t, H5J_{HH} 5.5 Hz), 5.7724 (3H, s, H7), 3.4108 (6H, q, H9 J_{HH} 6.3 Hz), 1.1142 (9H, d, H10 J_{HH} 7.1 Hz); NMR ^{13}C ($CDCl_3$, δ in ppm) 138.0390 (C2), 132.8175 (C3),129.9472 (C phen),128.3747 (C phen), 127.3059 (C phen), 123.0616 (C phen), 101.3817 (C7), 62.4648 (C9), 15.3035 (10).

3.2.2. Trans-dichloro-bis[tris(2-diethylacetal formyl phenyl)stibine] platinum (II) (**2**)

To a solution of stibine (**1**) (0.436 g, 1 mmol) made in 10 ml of acetone was added $Na_2[PtCl_4]$ (0.207 g, 0.5 mmol) dissolved in 10 ml of water. The resulting mixture was stirred for 12 h at room temperature and poured into 50 ml of water. The complex was extracted with chloroform (50 ml). The extract was dried over anhydrous sodium sulphate, concentrated to ~10 ml and mixed with hexane (20 ml). The resulting orange solid was filtered, washed with hexane and dried in vacuo. The single crystals of the complex **2** were grown from chloroform:hexane mixture (1:1). Yield: 1.04 g (66%); m.p.: 92–94 °C; IR (ν cm^{-1}): 476 (Sb–C), 2973 (C–H aromatic), 1056, 1091 (C–O–C) ; FAB^+ m/z 1577 (0.3%) $[M-4H]^+$, 1777 (1.5%) $[M+PtCl_2L]^+$, 179 (23%) $[L]^+$; NMR ^1H ($CDCl_3$, δ in ppm) 7.6954(6H, d, H3), 7.0819–7.6011 (18H, m, H4, H5, H6), 5.7037 (6H, s, H7), 3.4300 (12H, q, H9), 0.9540 (18H, t, H10); NMR ^{13}C ($CDCl_3$, δ in ppm) 144.3750 (C2), 140.0085 (C3), 137.8405 (C phen), 128.4357 (C phen), 127.0914 (C phen),

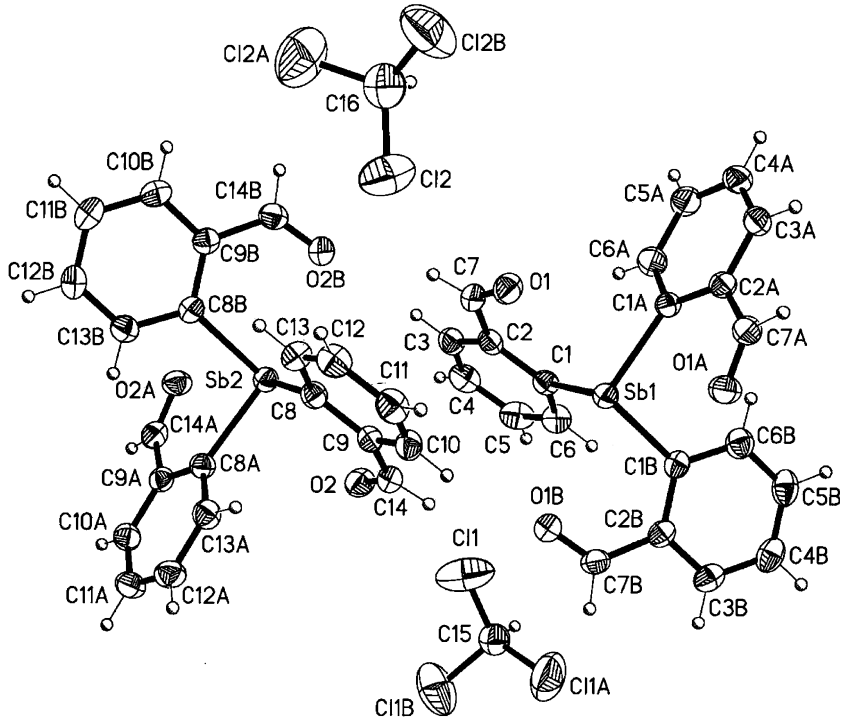

Fig. 2. Molecular structure of compound (**3**) in trigonal form.

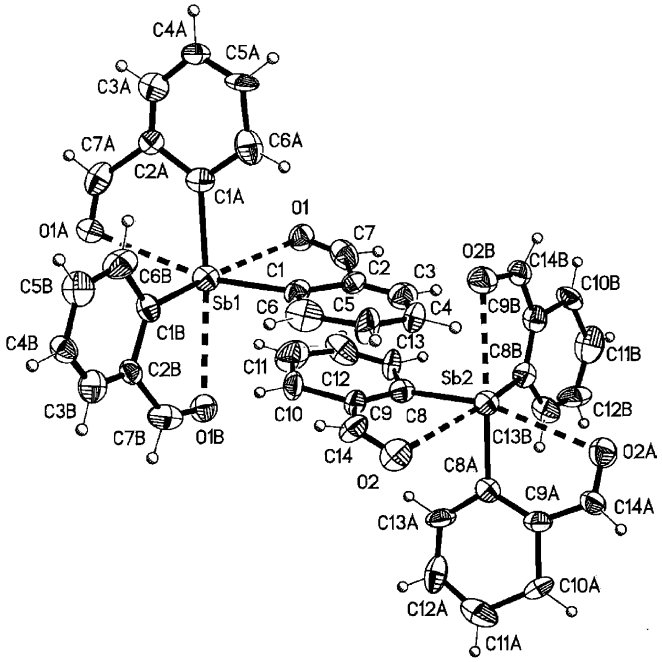

Fig. 3. Molecular structure of compound (**3**) in rhombohedral form.

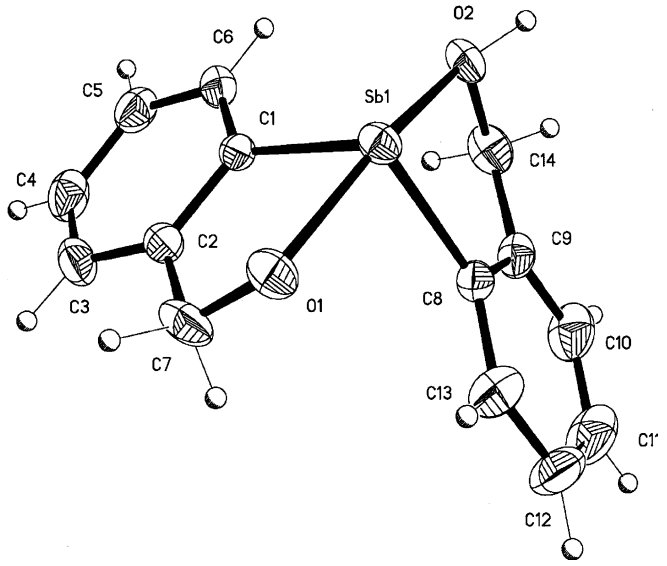

Fig. 4. Molecular structure of compound (**4**).

126.2525 (C phen), 102.7253 (C7), 61.3198 (C9), 14.8607 (10).

3.2.3. Tris (2-formylphenyl)stibine (*3*)

In a round bottom flask 1.974 g (3 mmol) of *tris*(2-diethylacetal formyl phenyl)stibine was dissolved in acetone, to this a 10 ml solution of 0.3 M HCl was added dropwise. After the addition was completed the reaction mixture was stirred for 4 h at room temperature. The compound was extracted with chloroform and the extract was washed with very dilute sodium carbonate solution and dried over sodium sulphate, concentrated to ~10 ml and mixed with hexane (20 ml). The resulting yellow solid was filtered and dried in vacuo. The single crystals of the compound were grown in toluene and chlorform.Yield: 0.390 g (84%); m.p.: 210–212 °C; Anal. Found (Calc.): C, 56.97 (57.79); H, 3.27 (3.44)% for $C_{21}H_{15}O_3Sb$; IR (ν cm^{-1}): 480 (Sb–C), 1694 (C=O), 2937 (C–H aromatic); NMR

^{1}H ($CDCl_3$, δ in ppm) 7.5289 (3H, d, H3), 7.2245 (6H, m, H4, H6), 7.0461 (3H, t, H5), 10.1413 (3H, s, H7); NMR ^{13}C ($CDCl_3$, δ in ppm) 137.3520 (C2), 135.0466 (C3), 133.7488 (C phen), 128.5884 (C phen), 127.9472 (C phen), 126.2677 (C phen), 194.6357 (C7).

*3.2.4. (2-Hydroxy methyl)phenyl[2-(3H-benzo[1,2] oxastibol) (**4**)*

In a 50 ml round bottom flask, a mixture of 1.308 g (3 mmol) of *tris*(2-benzaldehyde)stibine and sodium borohydride 0.5 g (12 mmol) in ethanol (30 ml) was stirred for 3 h at room temperature. The solution was concentrated, and water was added. The product was filtered and dried. After recrystallization from CH_2Cl_2:hexane (70:30) a colorless crystalline product was obtained Yield: 0.292 g (87%); m.p.: 184–185 °C; Anal. Found (Calc.): C, 49.74 (50.29); H, 3.37 (3.89)% for $C_{1413}H_{15}O_2Sb$; IR (ν cm^{-1}): 478 (Sb–C), 2930 (C–H), 3597 (OH); NMR ^{1}H ($CDCl_3$, δ in ppm) 7.9146 (1H, m, H3), 7.28–7.8 (3H, m, H4, H5, H6), 4.6135 (1H, s, H7), 4.9664 (2H, s, H8) 4.7441 (2H, s, H9); NMR ^{13}C ($CDCl_3$, δ in ppm) 136.8023 (C2), 133.4893 (C3), 130.8480 (C phen), 128.2525 (C phen), 128.1151 (C phen), 127.6571 (C phen), 63.8084 (C7), 65.7473 (C9).

4. Supplementary materials

Crystallographic data for the structural analyses have been deposited with the Cambridge Crystallographic Data Centre, CCDC Nos. 280709–280712 for compounds **2**, **3a**, **3b** and **4**. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ UK [fax. (int. code) +44(1223)336 033, or deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk.

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