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μ-Oxobridged heterotrinuclear compounds containing antimony(V) and arsenic(III). Crystal structures of $Ar_3Sb(\mu\text{-}OAsPh_2)_2$ (Ar = Ph or *p*-tol)

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Abstract

Novel compounds containing open chain $Sb(OAs)_2$ units have been synthesised by reacting Ar_3SbBr_2 (Ar = Ph or *p*-tol) with Ph_2AsLi in dry THF/benzene under a N_2 atmosphere, followed by a work up of the reaction mixture under ambient conditions. Their single crystal structures revealed that the geometry of antimony is trigonal bipyramidal and that of arsenic somewhat distorted pyramidal. The Sb···As distance is shorter than the sum of their van der Waals radii, but this does not severely distort the regular trigonal bipyramidal geometry of antimony. The ^1H- and ^{13}C-NMR spectra of these compounds are generally as expected. In the mass spectrum of $Ph_3Sb(\mu\text{-}OAsPh_2)_2$, the molecular ion peak was observed, but for the other compound, peaks of only its fragments were observed. The ν(Sb–O–As) appears at 744 cm^{-1} in IR spectra of these compounds. © 1999 Elsevier Science S.A. All rights reserved.

Keywords: Antimony; Arsenic; Crystal structure; Oxobridged compound

1. Introduction

There is current interest [1–8] in small clusters, cages and rings formed by antimony alone or with phosphorus and arsenic. The attractive features of these systems include the novel co-ordination geometry of antimony [5], the unusual ligation mode of groups [4] used as building blocks in these systems and the possible applications as CVD precursors [9] for III–V semiconductors. In two recent publications, a trinuclear antimony moiety has been synthesised, in one case as a cation [5] $[Me_2Sb\text{–}Sb(Me)_2\text{–}SbMe_2]^+$ and in another as a neutral entity $R'_3R_4Sb_3$ [7] (R = Ph or *p*-tol; R′ = Ph, *p*-tol or Me). It was therefore thought worthwhile to design an oxobridged triheteronuclear cluster $Ar_3Sb(\mu\text{-}OAsPh_2)_2$. These are the first examples containing a AsOSbOAs unit in which antimony is in oxidation state V and arsenic in III. In the literature, only a few reports on cage compounds [1] having Sb–O–Sb and Sb–O–P as units exist. Recently, compounds having oxobridged Sb(V) and As(V) have also been described [2]. An adduct of 10-chlorophenoxarsine oxide with $SbCl_5$ reported by Holliday et al. ([10]a) also has As–O–Sb units. In the structure of dimeric triphenylstibine oxide ([10]b) the presence of Sb–O–Sb units has also been established. The distance between antimony and arsenic in the present AsOSbOAs unit is shorter than the sum of the van der Waals radii of both Sb and As.

2. Experimental

All solvents were dried, made oxygen free and redistilled before use. Triphenylarsine (Merck), lithium metal (Merck) and 2-chloro-2-methylpropane (Fluka)

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were used as-received. The published method ([11]a) was used to prepare a deep red solution (in dry THF) of Ph_2AsLi. To synthesise Ph_3SbBr_2 ([11]b) and p-tol_3SbBr_2 [12], published methods were used. These compounds were stored under oxygen-free nitrogen.

C and H analyses were performed on a 240C Perkin-Elmer Analyser. The IR spectra were recorded on a Nicolet 5DX FT-IR spectrometer as KBr pellets. NMR spectra, ^1H (99.55 MHz) and ^{13}C (25.0 MHz), were recorded on a JEOL JNM FX-100 FT-NMR spectrometer using $SiMe_4$ as an internal standard. The FAB MS spectra were recorded on a JEOL JMS-SX 102A mass spectrometer using 3-NOBA as matrix.

2.1. Triphenylantimony(V)bis{(μ-oxo)-diphenylarsenic(III)}, $Ph_3Sb(OAsPh_2)_2$, **1**

Ph_3SbBr_2 (2.56 g, 5 mmol) dissolved in dry benzene (20 ml) was added dropwise to a solution of Ph_2AsLi (10 mmol) generated in situ in dry THF (freshly distilled) under an oxygen-free nitrogen atmosphere (15 ml). The deep red colour of mixture due to Ph_2AsLi faded to light yellow. Thereafter, the mixture was stirred for 30 min, the solvent was removed in vacuo and the pasty mass left was extracted with 50 ml of benzene. The undissolved LiBr and LiCl were filtered off from the benzene extract and it was concentrated to 10 ml. $Ph_3Sb(OAsPh_2)_2$ was obtained as a colourless crystalline solid by adding hexane (5 ml) to this concentrate. It was recrystallised from benzene:hexane (moisture-free) mixture (1:1) and dried in vacuo. Yield: 2.53 g, 60%, m.p. 144°C. Anal. Calc. for $C_{42}H_{35}As_2O_2Sb$: C, 59.82; H, 4.18. Found: C, 59.30; H, 4.03. ^1H-NMR (C_6D_6): δ 7.10 (m, 21H, ArH, *m* and *p* to As plus *m* and *p* to Sb), 7.60 (m, 8H, ArH, *o* to As), 8.30 (m, 6H, ArH, *o* to Sb). ^{13}C-NMR ($CDCl_3$): δ 150.9 (C *ipso* to Sb), 140.6 (C *ipso* to As), 137.2(C *o* to Sb and As), 132.9(C *p* to Sb), 131.0(C *m* to Sb), 130.1 (C *p* and C *m* to As). IR (KBr): 744 cm^{-1} (ν(Sb–O–As)). FAB MS: *m*/*z* 842, 844 (M), 840 (M-2H), 765, 767 (M-Ph), 686 (M-(Ph_2 + 2H)), 613, 615 (M-Ph_2As), 597, 599 (M-Ph_2AsO), 551 ($Ph_2SbSbPh_2$ + H), 520, 522 (M-(Ph_2AsO + Ph)), 504, 506 ($Ph_2SbAsPh_2$), 490 (M−Ph_3Sb), 474 (M-Ph_3SbO), 458 ($Ph_2AsAsPh_2$), 369, 371 (Ph_3SbO + H), 352, 354 (Ph_3Sb), 322 (Ph_2AsOPh), 306 (Ph_3As), 275, 277 (Ph_2Sb), 273, 275 ($C_{12}H_8Sb$), 245 (Ph_2AsO), 229 (Ph_2As), 227 ($PhAs_2$), 198, 200 (PhSb), 168 (PhAsO), 154 (Ph_2), 152 (PhAs), 137, 139 (SbO), 75 (As). The isotopic pattern of antimony was observed for strong peaks only.

2.2. Tri-*p*-tolylanitmony(V)bis{(μ-oxo)-diphenylarsenic(III)}, p-$tol_3Sb(OAsPh_2)_2$, **2**

Compound **2** was prepared by a method similar to that of **1**, except that p-tol_3SbBr_2 (2.77 g, 5 mmol) was used in place of Ph_3SbBr_2 and **2** was finally recrystallized from a chloroform:hexane mixture (1:1), as colourless crystals. Yield: 2.88 g, 65%, m.p. 176°C. Anal. Calc. for $C_{45}H_{41}As_2O_2Sb$: C, 61.05; H, 4.67. Found: C, 61.32; H, 4.64. ^1H-NMR (C_6D_6): δ 1.99 (s, 9H, CH_3), 7.10 (m, 18H, ArH, *m* to Sb plus *m* and *p* to As), 7.64 (m, 8H, ArH, *o* to As), 8.26 (m, 6H, ArH, *o* to Sb). ^{13}C-NMR ($CDCl_3$): δ 152.0 (C *p* to Sb), 143.6

Table 1

Crystallographic data, measurements and refinements of $Ph_3Sb(OAsPh_2)_2$, **1**, and p-$tol_3Sb(OAsPh_2)_2$, **2**

Compound	**1**	**2**
Empirical formula	$C_{42}H_{30}As_2O_2Sb$	$C_{45}H_{41}As_2O_2Sb$
Formula weight	838.3	885.4
Colour, habit	Colourless, irregular	Colourless, prism
Temperature (K)	298	293
Crystal system	Triclinic	Triclinic
Space group	$P\bar{1}$	$P\bar{1}$
Unit cell dimensions:		
a (Å)	10.136(2)	10.210(2)
b (Å)	14.002(2)	13.933(2)
c (Å)	14.287(2)	15.323(2)
α (°)	113.71(2)	107.18(2)
β (°)	96.45(2)	102.36(2)
γ (°)	93.33(2)	97.29(2)
V (Å^3)	1832.8(3)	1991.8(4)
Z	2	2
$D_{calc.}$ (Mg m^{-3})	1.519	1.476
Absorption coefficient (mm^{-1})	2.578	2.377
F(000)	830	888
Crystal size (mm^3)	0.56×0.4×0.3	0.42×0.24×0.10
Scan speed (° min^{-1} in ω)	Variable; 8.00–100.00	Variable; 4.00–100.00
Scan range, ω (°)	1.24	1.06
Index ranges	$0\leq h\leq 13$, $-16\leq k\leq 16$, $-18\leq l\leq 18$	$0\leq h\leq 13$, $-17\leq$ k≤ 16, $-19\leq l\leq 19$
Reflections collected	8587	10 354
Independent reflections	8128 (R_{int} = 3.41%)	9755 (R_{int} = 11.81%)
Min/max transmission	0.2973/0.3566	0.3162/0.4444
Quantity minimized	$\Sigma w(F_o-F_c)^2$	$\Sigma w(F_o-F_c)^2$
Extinction correction[a]	$\chi = -0.00014(7)$	$\chi = -0.00021(7)$
Number of parameters refined	415	452
Final R indices[b] (obs. data)	R = 5.56%, wR = 5.92%	R = 6.07%, wR = 6.17%
R indices (all data)	R = 12.42%, wR = 9.14%	R = 15.69%, wR = 8.61%
Goodness-of-fit	1.13	1.16
Largest, mean Δ/σ	2.043, 0.016	3.116, 0.009
Data-to-parameter ratio	9.7:1	9.0:1
Largest difference peak and hole (eÅ^{-3})	0.74, −0.79	0.80, −0.64

[a] $F^* = F[1+0.002\chi F^2/\sin(2\theta)]^{-1/4}$.

[b] Weighing scheme, $w^{-1} = \sigma^2(F)+0.0008F^2$.

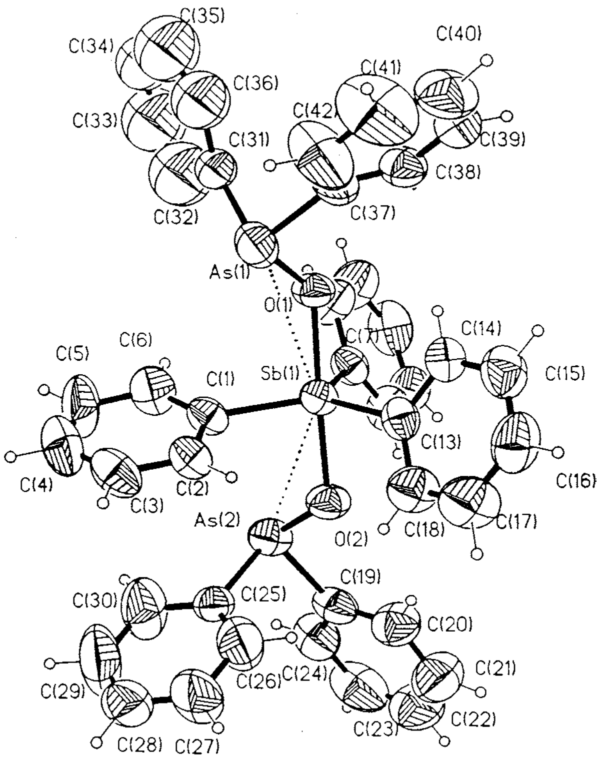

Fig. 1. Molecular structure (ORTEP diagram) of $Ph_3Sb(OAsPh_2)_2$, **1**.

(C *ipso* to As), 138.0 (C *ipso* to Sb), 133.6 (C *o* to Sb and As), 132.4 (C *m* to Sb), 131.0 (C *p* and C *m* to As) 23.0 (CH_3). IR (KBr): 744 cm^{-1} (ν(Sb–O–As)). FAB MS: m/z 716 (M-(p-tol + Ph)), 702 (M-(p-tol)$_2$), 655, 657 (M-Ph_2As), 639, 641 (M-Ph_2AsO), 548, 550 (M-(p-tol + Ph_2AsO)), 534 (M-((p-tol)$_2$ + PhAsO)), 487 (p-tol)$_3$SbOPh), 474 ($Ph_2AsOAsPh_2$), 411, 413 (p-tol$_3$SbO + H), 303, 305 (p-tol$_2$Sb), 245 (Ph_2AsO), 229 (Ph_2As), 154 (Ph_2), 137, 139 (SbO), 91 (p-tol). Only in strong peaks was the isotopic pattern of antimony noticed.

2.3. X-ray diffraction analysis

The colourless single crystals of **1** and **2** were grown from benzene:hexane (1:1) and chloroform:hexane(1:1) mixtures, respectively. The diffraction data were collected on a Siemens P4/PC system using graphite monochromated Mo–K$_\alpha$ radiation ($\lambda = 0.71073$ Å). The ω scan mode was used. The 2θ values were in the range 3.0°–55.0°. The background measurements were made for 25% of total scan time keeping the crystal and counter stationary at the beginning and end of the scan. Three standard reflections were measured after every 97 reflections. The semi-empirical absorption corrections were made. The SHELXTL PLUS (PC version) [13] program was used to refine the structures. The structure was solved by direct methods and for final refinement cycle the full-matrix least-squares method was used. The hydrogen atoms were included by the riding model (fixed isotropic U). The crystal data and other details of data collection, solution and refinement are given in Table 1.

3. Results and discussion

The Ph_2AsLi generated according to Eq. (1) on reaction with Ar_3SbBr_2, results in **1** or **2** (Eq. (2)), which are the first examples of compounds containing the $Sb(OAs)_2$ unit.

$$Ph_3As \xrightarrow[\text{(ii) } t-\text{BuCl/THF/N}_2]{\text{(i) 2Li/THF/N}_2} Ph_2AsLi + C_6H_6 + LiCl + (CH_3)_2C{=}CH_2 \tag{1}$$

$$2Ph_2AsLi + Ar_3SbBr_2 \xrightarrow[\text{2LiBr}]{\text{benzene/N}_2} Ar_3Sb(AsPh_2)_2 \xrightarrow{\text{air}} Ar_3Sb(OAsPh_2)_2 \tag{2}$$

$Ar = Ph$ or $p - tol$

It appears that the open chain triheteronuclear compound containing the $SbAs_2$ unit was the most probable intermediate formed by the method summarised in Eqs. (1) and (2) with the oxygen inserted into the Sb–As bond from air during the work up under ambient conditions. The insertion of oxygen (using molecular O_2) into a Sb=Sb bond has been used recently [14] to prepare a Sb–O–Sb unit. However, we failed to isolate the intermediate containing the As–Sb–As unit even after using very stringent conditions and Schlenk techniques. The compounds **1** and **2** are soluble in common organic solvents and authenticated by elemental analyses. They can be stored under ambient conditions for several months without any sign of decomposition.

3.1. Spectral data

The ^1H- and ^{13}C-NMR spectra of **1** and **2** are gen-

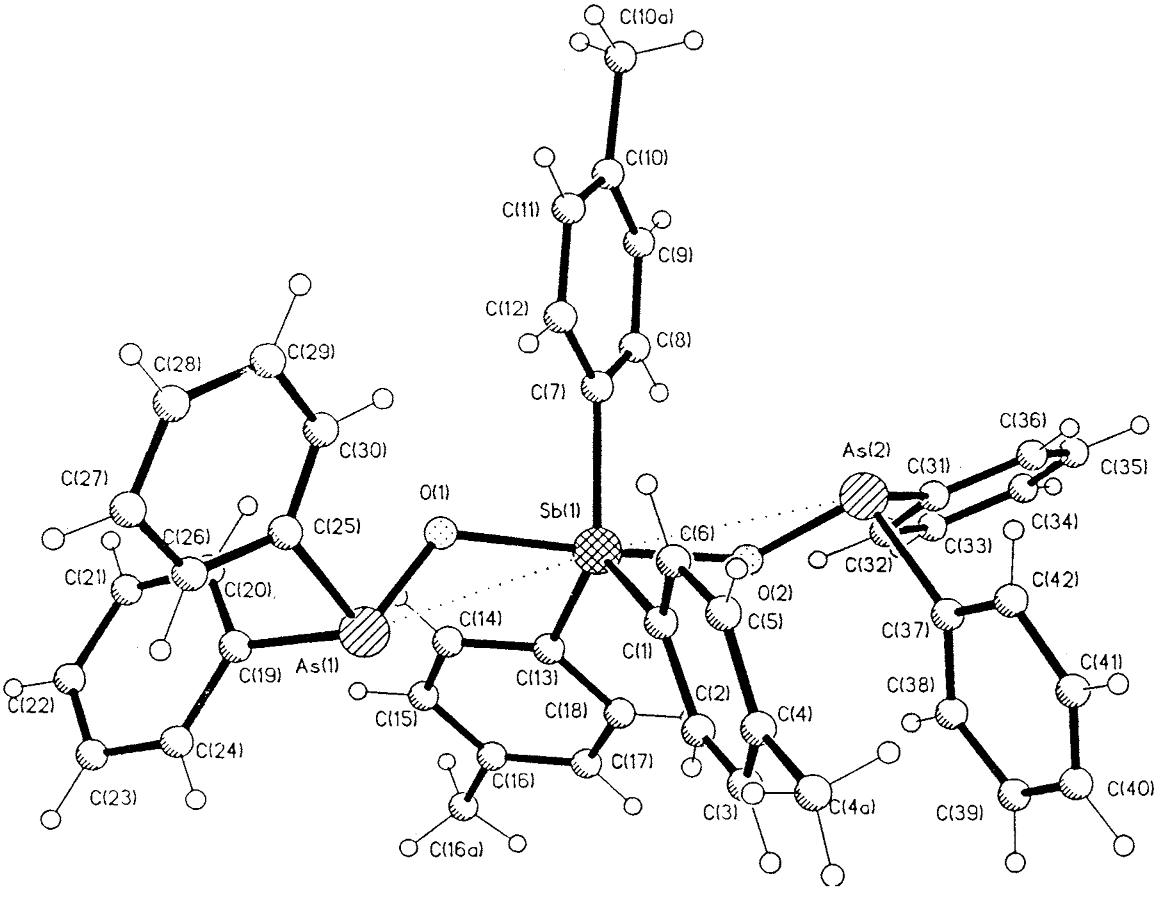

Fig. 2. Molecular structure (ORTEP diagram) of p-tol$_3$Sb(OAsPh$_2$)$_2$, **2**.

erally as expected. The ^{13}C-NMR assignments are based on the literature reports [12]. In the FAB MS spectrum of **1**, the molecular ion peak was observed, but was not observed in that of **2**. However, there are peaks in the mass spectrum of **2** that may be assigned to its fragments, e.g. (M-(p-tol)$_2$), (M-(p-tol + Ph$_2$AsO)) and (M-Ph$_2$As), supporting indirectly the molecular stoichiometry of **2**. In the IR spectra of **1** and **2**, a band appearing at 744 cm^{-1} was not noticed in the spectra of Ph$_3$Sb, Ph$_3$SbBr$_2$, p-tol$_3$Sb, p-tol$_3$SbBr$_2$, Ph$_3$As. Moreover, ν_{as}(As–O–As) is reported in [15] at 920 cm^{-1} in the IR spectrum of Me$_2$As(S)OAs(S)Me$_2$ and ν_{as}(Sb–O–Sb) is assigned to a band ([10]b) appearing at 658/664 cm^{-1} in the IR spectrum of triphenylstibine oxide dimer. Consequently, it is reasonable to conclude that the band appearing at 744 cm^{-1} most probably originates from the asymmetric vibrations of the Sb–O–As unit.

3.1.1. Crystal structures

The atomic co-ordinates for **1** and **2** along with equivalent isotropic displacement coefficients are available as supplementary material. The ORTEP diagrams (molecular structures) are shown in Figs. 1 and 2. The selected bond lengths and bond angles are given in Table 2. The crystal of **1** was not of good quality, which resulted in disorder in one of the phenyl rings. The arrangement of donor atoms of various ligands around antimony is nearly a regular trigonal bipyramidal. The geometry of arsenic is somewhat distorted pyramidal. The distances between arsenic and antimony in **1** and **2** are within the range 3.330–3.418 Å, which is significantly less than the sum of their van der Waals radii (4.210 Å) [16]. This may be interpreted in terms of intramolecular secondary interaction between arsenic and antimony. Some role of this secondary intramolecular linkage in stabilising the μ-oxobridged Sb(OAs)$_2$ unit of **1** and **2** may be possible but in the structure of either of these two compounds, the proposed secondary linkage does not severely distort the geometry of Sb as expected for a hepta-co-ordination. So far only one authentic report on the VII co-ordination of Sb is available [17] in the literature. The C–C distances and C–C–C bond angles of phenyl rings (1.250(38)–1.442(26) Å and 110.1(22)–129.8(20)°, respectively) are normal. The Sb–O and As–O bond lengths are in the ranges 2.041(7)–2.054(7) and 1.758(6)–1.781(5) Å, respectively, and comparable with the earlier reported As/Sb–O single bond length [18]. However, the present As–O bond lengths are longer than those reported [2]

Table 2
Selected bond lengths (Å) and angles(°) with estimated S.D.s in parentheses

Compound	**1**	**2**
Bond length (Å)		
Sb(1)–O(1)	2.041(7)	2.051(8)
Sb(1)–O(2)	2.051(7)	2.054(7)
Sb(1)–C(1)	2.112(9)	2.123(12)
Sb(1)–C(7)	2.112(10)	2.101(10)
Sb(1)–C(13)	2.108(9)	2.117(9)
As(1)–O(1)	1.758(6)	1.768(7)
As(1)–C(37)	1.969(9)	–
As(1)–C(31)	1.777(23)	–
As(1)–C(31A)	2.326(31)	–
As(2)–O(2)	1.781(5)	1.762(5)
As(2)–C(19)	1.961(13)	–
As(2)–C(25)	1.952(11)	–
As(1)–C(19)	–	1.969(9)
As(1)–C(25)	–	1.967(14)
As(2)–C(31)	–	1.972(14)
As(2)–C(37)	–	1.964(13)
Sb(1)···As(1)	3.418	3.389
Sb(1)···As(2)	3.330	3.373
Bond angles (°)		
O(1)–Sb(1)–O(2)	177.5(3)	176.5(3)
O(1)–Sb(1)–C(1)	92.7(3)	93.3(4)
O(2)–Sb(1)–C(1)	89.7(3)	89.2(4)
O(1)–Sb(1)–C(7)	87.0(3)	86.1(3)
O(2)–Sb(1)–C(7)	91.3(3)	90.5(4)
C(1)–Sb(1)–C(7)	122.6(3)	120.3(4)
O(1)–Sb(1)–C(13)	92.6(3)	94.4(4)
O(2)–Sb(1)–C(13)	86.7(3)	86.6(3)
C(1)–Sb(1)–C(13)	117.1(3)	118.3(4)
C(7)–Sb(1)–C(13)	120.3(3)	121.3(4)
O(1)–As(1)–C(37)	99.2(4)	–
O(1)–As(1)–C(31)	108.5(7)	–
C(37)–As(1)–C(31)	94.0(7)	–
O(1)–As(1)–C(31A)	83.6(8)	–
C(37)–As(1)–C(31A)	97.2(7)	–
C(31)–As(1)–C(31A)	24.9(10)	–
O(2)–As(2)–C(19)	96.6(4)	–
O(2)–As(2)–C(25)	99.7(3)	–
C(19)–As(2)–C(25)	96.0(5)	–
Sb(1)–O(1)–As(1)	128.1(4)	124.9(4)
Sb(1)–O(2)–As(2)	120.7(4)	124.1(4)
O(1)–As(1)–C(19)	–	100.3(4)
O(1)–As(1)–C(25)	–	98.0(5)
C(19)–As(1)–C(25)	–	95.2(5)
O(2)–As(2)–C(31)	–	95.9(4)
O(2)–As(2)–C(37)	–	101.0(4)
C(31)–As(2)–C(37)	–	97.6(6)

(1.701(3)–1.711(3) Å), for $[(SbPh_2)_2(\mu\text{-}O)_2(\mu\text{-}O_2AsMe_2)_2]\cdot 2CHCl_3$ and $[\{Sb(p\text{-}MeC_6H_4)_2\}_2(\mu\text{-}O)_2(\mu\text{-}O_2AsMe_2)_2]$. The bond lengths of Sb–C (2.101(10)–2.117(9) Å) and As–C (1.777(23)–1.972(14) Å) observed in the structures of **1** and **2** are similar to those reported for Sb/As–C single bond [18,19] (2.090(13)–2.175(6) Å/1.820(3)–1.911(4) Å). The Sb–O–As bond angles (120.7(4)–128.1(4)°) are larger than the values (119.0(1)–119.5(1)°) reported recently [2] for $[(SbAr_2)_2(\mu\text{-}O)_2(\mu\text{-}O_2AsMe_2)_2]$. This may be due to strong steric effects of bulky aryl groups attached to antimony and arsenic both.

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