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High metal coordination numbers in group 15 organometallics: crystal structures of triphenylbismuthbis(trifluoroacetate) and triphenylantimonybis(trifluoroacetate)

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Abstract

Crystals of $Ph_3Bi(OCOCF_3)_2$ have been shown by X-ray diffractometry to be made up of isolated molecules in which the coordination at bismuth is intermediate between trigonal bipyramidal, with apical monodentate CF_3COO groups and pentagonal bipyramidal with equatorial bidentate CF_3COO groups. In the case of $Ph_3Sb(OCOCF_3)_2$ there is essentially trigonal bipyramidal coordination at antimony, with apical CF_3COO groups, and with a quite different conformation from that found for the bismuth compound.

Introduction

Recent structural studies [1,2] of the arsenic and antimony ylides **1–4** have shown the presence of attractive, intra-molecular M ··· O (M = As, Sb) interactions of the type indicated in **7** and **8**: the Sb ··· O interactions are much stronger than the As ··· O interactions, but in both types of ylide the overall geometry at arsenic or antimony is intermediate between tetrahedral and trigonal bipyramidal. Although the analogous bismuth ylides **5** and **6** are known [3,4], their extreme insolubility in most solvents prevented generation of crystals suitable for an X-ray

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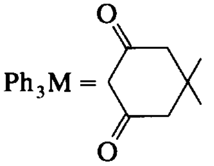
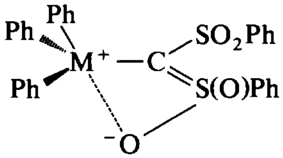
study. We have therefore been unable to compare the tendency of bismuth to form intra-molecular M · · · O interactions with those of its lighter congeners.

$Ph_3M{=}C(SO_2Ph)_2$

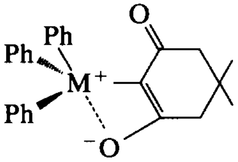
(**1**: M = As;
3: M = Sb;
5: M = Bi)



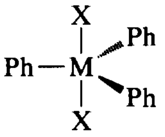
(**2**: M = As;
4: M = Sb;
6: M = Bi)



(**7**)



(**8**)



$X{=}CF_3C(O)O$

(**9**: M = Bi;
10: M = Sb)

Such comparison between bismuth and antimony can however be made using the formally five-coordinate compounds $Ph_3M(OCOCF_3)_2$ (**9**, M = Bi and **10**, M = Sb). A further point of interest is the possibility that five-coordinate group 15 atoms can adopt square-pyramidal (or rectangular pyramidal) geometry; while this is most commonly observed in the presence of cyclic ligands [5–9], some examples are known ($SbPh_5$ [10] and $BiPh_5$ [11]) in which solely monodentate ligands are present. While square (rectangular) pyramidal coordination is well established for both arsenic and antimony (and in group 14 for both germanium [12] and tin [13]), it has not yet been reported for either lead or bismuth.

We report below the X-ray crystal and molecular structures of compounds **9** and **10**.

Experimental

Compounds **9** and **10** were made by reaction of the appropriate dibromide, Ph_3MBr_2, with silver trifluoroacetate [14,15]. Crystals suitable for X-ray examination were grown from solutions in light petroleum (b.p. 40–60 °C).

Crystal data

9, $C_{22}H_{15}BiF_6O_4$, $M_r = 666.32$. Orthorhombic, $a = 13.630(2)$, $b = 16.524(4)$, $c = 20.240(6)$ Å, $V = 4559(2)$ Å^3. Space group *Pbca* (No. 61) (uniquely from systematic absences, $0kl$: $k = 2n + 1$; $h0l$: $l = 2n + 1$; $hk0$: $h = 2n + 1$). $Z = 8$, $D_x = 1.942$ g cm^{-3}, $\mu(\text{Mo-}K_\alpha) = 77.84$ cm^{-1}, $\lambda = 0.71073$Å, $F(000) = 2528$.

10, $C_{22}H_{15}F_6O_4Sb$, $M_r = 579.10$. Monoclinic, $a = 8.916(2)$, $b = 9.885(2)$, $c = 13.014(2)$ Å, $\beta = 99.08(1)°$, $V = 1132.6(4)$ Å^3. Space group *Pn* or *P*2/*n* from systematic absences ($h0l$: $l = 2n + 1$); both were considered, only *Pn* (No. 7) gave satisfactory analysis. $Z = 2$, $D_x = 1.698$ g cm^{-3}, $\mu(\text{Mo-}K_\alpha) = 12.96$ cm^{-1}, $\lambda = 0.71073$ Å, $F(000) = 568$.

Data collection

For both compounds accurate cell dimensions and crystal orientation matrices were determined with a CAD4 diffractometer by least-squares treatment of the setting angles of 25 reflections in the range $10° \leqslant \theta \leqslant 17.5°$. Data were collected in the $\omega-2\theta$ scan mode, using graphite-monochromated Mo-K_α radiation. Details of crystal size and reflections measured are given in Table 1. Data were corrected for Lorentz, polarisation, and absorption effects.

Structure solution and refinement

Patterson methods were used to determine the coordinates of Bi in **9** and Sb in **10**; other non-hydrogen atoms were found by the heavy-atom method. Refinement was by full-matrix least-squares calculations. All non-hydrogen atoms were allowed anisotropic thermal parameters; the atoms of the phenyl rings were refined as rigid groups as regular hexagon rings with C–C 1.395 Å (all hydrogen atoms were clearly visible in difference maps (see deposition material)). Details of the refinement, R-factors etc. are in Table 1. Final difference maps were featureless. The space group Pn for **10** is polar and a parallel calculation with the polar direction reversed gave R factors which were slightly higher (0.027 compared with 0.023 for R, 0.037 compared with 0.031 for wR), confirming that the correct direction of the polar axis had been selected. All calculations were performed on a Silicon Graphics 4D-380 computer using a Unix version of NRCVAX [16]; scattering factors and anomalous dispersion corrections were from refs. 17–19.

Atomic coordinates for **9** and **10** are in Tables 2 and 3, respectively. In each structure the asymmetric unit contains one formula unit and no crystallographic symmetry is required. The bond lengths and angles of the non-phenyl atoms are given in Tables 4 and 5; perspective views of **9** and **10** are in Figs. 1 and 2 respectively.

Additional material submitted to the referees and available from either the authors or the Cambridge Crystallographic Data Centre comprises H-atom coordi-

Table 1

Details of the X-ray analyses

	9	**10**
Crystal dimensions (mm)	0.63×0.38×0.18	0.50×0.26×0.12
Range of reflections measured		
h	0 to 17	−11 to 11
k	0 to 21	0 to 12
l	0 to 25	0 to 16
No. of reflections measured	4953	2726
No. of unique reflections	4953	2616
Reflections with $I > 2.5\sigma(I)$	1973	2342
Max. and min. transmission coeffs. in abs. correction	0.274 0.069	0.857 0.730
p in $w = 1/[\sigma^2(F_o) + p(F_o)^2]$	0.0006	0.0006
R	0.028	0.023
w^r	0.037	0.031
Density in final Δ-map	−0.64 to +0.63	−0.25 to +0.35
Shift/error in last LS cycle	0.021	0.054

Table 2

Fractional atomic coordinates and equivalent isotropic thermal parameters B_{eq} (Å^2) for **9**

	x	y	z	B_{eq} [a]
Bi	0.38005(2)	0.00068(3)	0.32155(1)	3.26(2)
F1	0.5851(8)	−0.2423(6)	0.3365(8)	15.4(11)
F2	0.6875(6)	−0.1614(6)	0.3535(7)	14.5(9)
F3	0.6139(11)	−0.2148(10)	0.4281(5)	21.3(11)
F4	0.0609(5)	0.1076(4)	0.3802(5)	10.4(6)
F5	0.1270(6)	0.2164(5)	0.3536(5)	8.7(5)
F6	0.1263(6)	0.1817(6)	0.4524(4)	10.3(6)
O1	0.5012(5)	−0.0963(4)	0.3130(4)	4.4(4)
O2	0.5006(6)	−0.0847(4)	0.4226(4)	5.0(4)
O3	0.2523(6)	0.0925(4)	0.3226(4)	4.2(3)
O4	0.2770(6)	0.0847(4)	0.4319(4)	4.8(4)
C1	0.5291(9)	−0.1156(7)	0.3712(6)	4.4(6)
C2	0.6060(10)	−0.1822(9)	0.3744(10)	6.5(9)
C3	0.2324(9)	0.1065(6)	0.3836(6)	3.7(5)
C4	0.1358(9)	0.1522(7)	0.3914(7)	5.1(6)
C11	0.3683(5)	−0.0027(5)	0.2139(2)	4.0(4)
C12	0.4351(4)	−0.0477(4)	0.1768(4)	4.7(5)
C13	0.4229(5)	−0.0549(4)	0.1086(4)	5.7(6)
C14	0.3438(6)	−0.0171(5)	0.0776(3)	6.4(7)
C15	0.2771(5)	0.0279(5)	0.1147(4)	7.9(8)
C16	0.2893(4)	0.0352(5)	0.1828(4)	6.6(7)
C21	0.4822(4)	0.0920(4)	0.3510(4)	3.5(5)
C22	0.5280(5)	0.1360(5)	0.3009(3)	5.0(6)
C23	0.5939(5)	0.1976(5)	0.3168(4)	6.2(7)
C24	0.6140(4)	0.2152(4)	0.3828(4)	5.3(6)
C25	0.5682(5)	0.1712(4)	0.4329(3)	4.6(5)
C26	0.5023(5)	0.1096(4)	0.4170(3)	3.7(5)
C31	0.2810(5)	−0.0868(4)	0.3634(4)	3.6(5)
C32	0.2241(6)	−0.1308(5)	0.3188(3)	5.8(7)
C33	0.1569(5)	−0.1879(4)	0.3420(4)	6.8(8)
C34	0.1466(5)	−0.2008(4)	0.4097(5)	6.3(8)
C35	0.2035(6)	−0.1568(5)	0.4543(3)	6.1(7)
C36	0.2707(5)	−0.0997(4)	0.4311(4)	4.5(6)

[a] B_{eq} is the mean of the principal axes of the thermal ellipsoid.

nates, thermal parameters, torsion angles, mean plane data, structure factor listings and difference maps showing the phenyl H atoms.

Results and discussion

$Ph_3Bi(OCOCF_3)_2$

The structure of $Ph_3Bi(OCOCF_3)_2$ consists of isolated molecules which have approximate C_{2v} ($mm2$) molecular symmetry, although no symmetry is imposed crystallographically. The coordination polyhedron defined by the atoms directly bonding to bismuth, namely C_3BiO_2, is a distorted trigonal bipyramid, in which the phenyl ligands occupy equatorial sites with the C_3Bi fragment essentially planar, while the trifluoroacetate ligands, expected to be the more apicophilic, occupy the axial sites. The phenyl ring conformations are defined by the dihedral angles which the ring planes make with the BiC_3 plane; two rings [C(21)–C(26) and C(31)–C(36)]

Table 3

Fractional atomic coordinates and equivalent isotropic thermal parameters B_{eq} (Å^2) for **10**

	x	y	z	B_{eq} [a]
Sb	0.0	0.83387(2)	0.0	2.84(1)
F1	0.7366(10)	0.8036(14)	1.1161(6)	15.2(9)
F2	0.5157(14)	0.8702(10)	1.0573(7)	11.7(6)
F3	0.5568(13)	0.6663(6)	1.0969(6)	11.6(5)
F4	0.8352(14)	0.7246(9)	0.4028(8)	14.3(6)
F5	1.0036(8)	0.8590(7)	0.4507(7)	8.1(4)
F6	0.8088(9)	0.9154(12)	0.3536(5)	13.8(6)
O1	0.6715(10)	0.8483(4)	0.8978(7)	3.7(2)
O2	0.6470(6)	0.6248(5)	0.9165(4)	5.6(2)
O3	0.8439(10)	0.8150(5)	0.6112(7)	3.7(2)
O4	0.6637(5)	0.9406(5)	0.5153(3)	5.1(2)
C1	0.6459(6)	0.7388(6)	0.9453(4)	3.9(2)
C2	0.6114(11)	0.7723(13)	1.0552(6)	6.5(4)
C3	0.7779(6)	0.8749(5)	0.5267(4)	3.8(2)
C4	0.8564(12)	0.8464(9)	0.4320(7)	5.4(3)
C11	0.7548(8)	1.0440(2)	0.7545(6)	3.4(1)
C12	0.8820(7)	1.1101(5)	0.8078(6)	6.0(4)
C13	0.8789(7)	1.2497(5)	0.8227(5)	7.5(5)
C14	0.7486(8)	1.3234(2)	0.7842(4)	6.2(4)
C15	0.6214(6)	1.2573(4)	0.7309(4)	5.4(3)
C16	0.6245(7)	1.1176(4)	0.7160(5)	4.6(3)
C21	0.5500(3)	0.7420(4)	0.6809(3)	3.2(3)
C22	0.4114(4)	0.8002(4)	0.6930(3)	4.3(2)
C23	0.2761(3)	0.7468(5)	0.6400(4)	5.3(3)
C24	0.2795(3)	0.6351(5)	0.5749(4)	5.4(3)
C25	0.4181(4)	0.5769(4)	0.5627(3)	5.2(3)
C26	0.5534(3)	0.6303(4)	0.6157(3)	4.4(2)
C31	0.9443(3)	0.7294(3)	0.8186(3)	3.2(3)
C32	1.0241(4)	0.7743(3)	0.9134(3)	4.2(2)
C33	1.1565(4)	0.7080(4)	0.9583(3)	5.0(3)
C34	1.2091(4)	0.5968(4)	0.9084(3)	4.9(3)
C35	1.1294(4)	0.5519(4)	0.8137(3)	4.8(2)
C36	0.9970(4)	0.6182(4)	0.7688(2)	4.3(2)

[a] B_{eq} is the mean of the principal axes of the thermal ellipsoid.

are almost coplanar with the BiC_3 plane (dihedral angles 2.0(3) and 2.1(3)°, respectively) while the C(11)–C(16) ring makes a dihedral angle of 98.5(2)° with the BiC_3 plane. In the equatorial plane of this polyhedron, the C(21)–Bi–C(31) angle of 140.6(3)° is much larger than the expected 120°, while the two other bond angles involving C(11) are correspondingly smaller than expected.

The formally non-bonded bismuth–oxygen distances Bi–O(2) and Bi–O(4) are only ca. 30% longer than the bonded distances Bi–O(1) and Bi–O(3): since the atoms C(11), O(1)–O(4) and Bi are nearly coplanar the bismuth coordination might equally well be viewed as a distorted pentagonal bipyramid in which the equatorial sites are occupied by C(11) and the four oxygen atoms O(1)–O(4), with C(21) and C(31) occupying the two axial sites: the bond angles in the equatorial plane of this polyhedron are shown in Fig. 3.

The Bi–C bond distances, ranging from 2.138(6) to 2.185(6) Å are comparable with those in the Ph_4Bi^+ cations found in several salts: $Ph_4Bi^+ClO_4^-$ [20],

Table 4

Molecular dimensions for **9**

a. Bond lengths (Å)			
Bi-O(1)	2.308(7)	F(5)-C(4)	1.315(16)
Bi-O(3)	2.309(7)	F(6)-C(4)	1.333(15)
Bi-C(11)	2.185(5)	O(1)-C(1)	1.279(14)
Bi-C(21)	2.138(6)	O(2)-C(1)	1.222(15)
Bi-C(31)	2.152(7)	O(3)-C(3)	1.285(15)
F(1)-C(2)	1.287(22)	O(4)-C(3)	1.206(14)
F(2)-C(2)	1.237(18)	C(1)-C(2)	1.522(17)
F(3)-C(2)	1.217(20)	C(3)-C(4)	1.525(16)
F(4)-C(4)	1.280(14)		
b. Bond angles (°)			
O(1)-Bi-O(3)	175.1(3)	F(2)-C(2)-F(3)	110.5(15)
O(1)-Bi-C(11)	87.7(3)	F(2)-C(2)-C(1)	113.7(12)
O(1)-Bi-C(21)	92.6(3)	F(3)-C(2)-C(1)	114.8(14)
O(1)-Bi-C(31)	90.7(3)	O(3)-C(3)-O(4)	128.1(11)
O(3)-Bi-C(11)	88.3(3)	O(3)-C(3)-C(4)	111.8(10)
O(3)-Bi-C(21)	91.43(24)	O(4)-C(3)-C(4)	120.0(11)
O(3)-Bi-C(31)	87.95(24)	F(4)-C(4)-F(5)	106.8(11)
C(11)-Bi-C(21)	110.1(3)	F(4)-C(4)-F(6)	107.4(11)
C(11)-Bi-C(31)	109.2(3)	F(4)-C(4)-C(3)	112.6(9)
C(21)-Bi-C(31)	140.6(3)	F(5)-C(4)-F(6)	103.6(10)
Bi-O(1)-C(1)	108.4(7)	F(5)-C(4)-C(3)	114.7(10)
Bi-O(3)-C(3)	106.7(7)	F(6)-C(4)-C(3)	111.1(11)
O(1)-C(1)-O(2)	125.9(11)	Bi-C(11)-C(12)	120.2(5)
O(1)-C(1)-C(2)	115.1(12)	Bi-C(11)-C(16)	119.6(5)
O(2)-C(1)-C(2)	119.0(12)	Bi-C(21)-C(22)	117.2(5)
F(1)-C(2)-F(2)	102.0(15)	Bi-C(21)-C(26)	122.8(5)
F(1)-C(2)-F(3)	102.2(15)	Bi-C(31)-C(32)	116.4(6)
F(1)-C(2)-C(1)	112.4(13)	Bi-C(31)-C(36)	123.6(5)

$Ph_4Bi^+CH_3C_6H_4SO_3^-$ [21], and $Ph_4Bi^+[Ph_2Bi(OCOCF_3)_2]^-$ [21]. These distances are, however, rather shorter than those in the anion $[Ph_2Bi(OCOCF_3)_2]^-$, 2.24(2) and 2.26(2) Å [21], and in the neutral square-pyramidal $BiPh_5$ [11] where the axial Bi–C distance is 2.221(9) Å, and the basal distances range from 2.322(9) to 2.329(9) Å. The Bi–O bonded distances are 2.308(7) and 2.309(7) Å while the corresponding non-bonded Bi–O distances are 2.980 and 2.981 Å giving ratios in each case of 1.29: in the only analogous system, the anion $[Ph_2Bi(OCOCF_3)_2]^-$ [21], the bonded and non-bonded distance are both longer (and less precise) than those found here with a ratio of non-bonded/bonded distances of 1.33.

The longer Bi–O distances in the anion $[Ph_2Bi(OCOCF_3)_2]^-$ are probably a consequence of the overall negative charge: however, in any such system, the ratio of non-bonded to bonded metal–oxygen distances is a measure of the degree to which the carboxylate becomes bidentate; in a symmetrically bidentate carboxylate ligand, this ratio becomes unity. By this criterion the trifluoroacetate ligands are much more nearly bidentate in $Ph_3Bi(OCOCF_3)_2$ and $[Ph_2Bi(OCOCF_3)_2]^-$ than in the antimony analogue $Ph_3Sb(OCOCF_3)_2$ (see below).

Table 5

Molecular dimensions for **10**

a. Bond lengths (Å)			
Sb-O(1)	2.153(9)	F(5)-C(4)	1.303(13)
Sb-O(3)	2.116(9)	F(6)-C(4)	1.244(12)
Sb-C(11)	2.0783(22)	O(1)-C(1)	1.284(8)
Sb-C(21)	2.076(3)	O(2)-C(1)	1.188(8)
Sb-C(31)	2.092(3)	O(3)-C(3)	1.305(10)
F(1)-C(2)	1.300(13)	O(4)-C(3)	1.197(7)
F(2)-C(2)	1.293(13)	C(1)-C(2)	1.546(10)
F(3)-C(2)	1.308(14)	C(3)-C(4)	1.536(10)
F(4)-C(4)	1.268(11)		
b. Bond angles (°)			
O(1)-Sb-O(3)	175.5(3)	F(2)-C(2)-F(3)	107.5(9)
O(1)-Sb-C(11)	85.25(24)	F(2)-C(2)-C(1)	114.1(7)
O(1)-Sb-C(21)	92.2(3)	F(3)-C(2)-C(1)	110.7(10)
O(1)-Sb-C(31)	90.87(24)	O(3)-C(3)-O(4)	127.8(6)
O(3)-Sb-C(11)	95.9(3)	O(3)-C(3)-C(4)	113.0(7)
O(3)-Sb-C(21)	91.16(25)	O(4)-C(3)-C(4)	119.1(6)
O(3)-Sb-C(31)	84.74(23)	F(4)-C(4)-F(5)	104.2(10)
C(11)-Sb-C(21)	117.52(21)	F(4)-C(4)-F(6)	105.1(10)
C(11)-Sb-C(31)	117.99(21)	F(4)-C(4)-C(3)	110.4(7)
C(21)-Sb-C(31)	124.47(13)	F(5)-C(4)-F(6)	107.7(8)
Sb-O(1)-C(1)	118.8(5)	F(5)-C(4)-C(3)	113.9(8)
Sb-O(3)-C(3)	119.5(6)	F(6)-C(4)-C(3)	114.6(9)
O(1)-C(1)-O(2)	129.5(6)	Sb-C(11)-C(12)	119.6(4)
O(1)-C(1)-C(2)	109.9(7)	Sb-C(11)-C(16)	119.9(4)
O(2)-C(1)-C(2)	120.5(7)	Sb-C(21)-C(22)	119.1(3)
F(1)-C(2)-F(2)	108.4(13)	Sb-C(21)-C(26)	120.68(22)
F(1)-C(2)-F(3)	106.1(9)	Sb-C(31)-C(32)	119.5(3)
F(1)-C(2)-C(1)	109.7(7)	Sb-C(31)-C(36)	120.47(23)

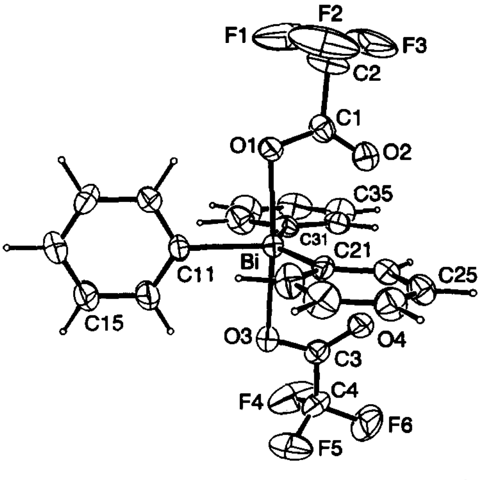


Fig. 1. Perspective view of the molecule of $Ph_3Bi(OCOCF_3)_2$, showing the atom-numbering scheme.

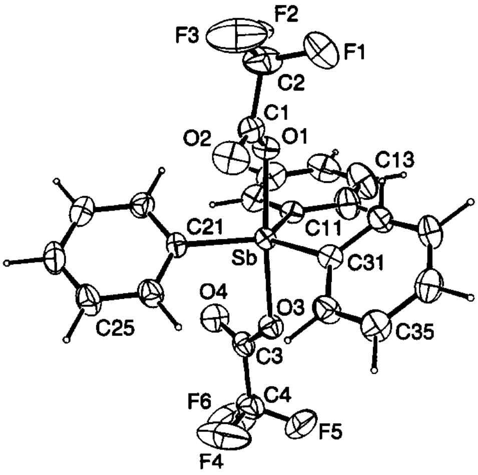


Fig. 2. Perspective view of the molecule of $Ph_3Sb(OCOCF_3)_2$, showing the atom-numbering scheme.

$Ph_3Sb(OCOCF_3)_2$

The structure of **10** also contains isolated molecules, but with a totally different conformation from that found for the bismuth derivative **9**. In **10** the Sb coordination is still distorted trigonal bipyramidal with the SbC_3 coordination group close to being planar, and only one of the phenyl rings [C(11)–C(16)] lies close to the SbC_3 plane (dihedral angle 8.1(2)°, corresponding to a bend in the C(14) · · · C(11)–Sb angle rather than a simple rotation about the Sb–C(11) bond); the other two ring planes [C(21)–C(26) and C(31)–C(36)] make dihedral angles with the SbC_3 plane of 40.3(2)° and −43.3(2)°, respectively. The CF_3CO_2 groups also take up different conformations quantified by the torsion angles C(11)–Sb–O(1)–C(1) (−178.6(6)°) and C(11)–Sb–O(3)–C(3) (−56.6(4)°). In $Ph_3Sb(OCOCH_3)_2$ [22] both CH_3CO_2 groups adopt a conformation similar to that in **9** with C–Sb–O–C torsion angles close to 180°.

The Sb–C bond lengths (Table 5) are somewhat shorter than those of 2.10–2.15 Å typically observed for phenyl groups bound to trigonal bipyramidal antimony [22,23], and just shorter even than that 2.095(2) Å found in the isolated tetrahedral

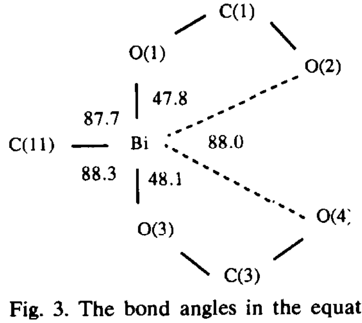


Fig. 3. The bond angles in the equatorial plane of $Ph_3Bi(OCOCF_3)_2$, when viewed as a pentagonal bipyramid.

cation in $Ph_4Sb^+ClO_4^-$ [23]. Antimony–oxygen bond lengths in organoantimony compounds are extremely variable [23], ranging from 1.935 Å in $(Ph_3SbO)_2$ [24] to 2.506 Å in Ph_4SbOSO_2Ph [25], and the Sb–O distances in **10** (2.116(9) and 2.153(9) Å) lie within this range.

The non-bonded Sb···O distances are 3.209 and 3.231 Å, giving ratios of non-bonded/bonded Sb–O distances of 1.52 and 1.50, larger than in the bismuth analogue described above and in the bis-acetate $Ph_3Sb(OCOCH_3)_2$ where the ratio is 1.31 [22]. This indicates a very asymmetric binding of the trifluoroacetate ligands in $Ph_3Sb(OCOCF_3)_2$ with effectively no interaction between the metal atom and the carbonyl oxygen atoms.

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