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Synthesis and *in vitro* antitumor activity of some tetraphenylantimony derivatives of *exo*-7-oxa-bicyclo[2,2,1] heptane (ene)-3-arylamide-2-acid

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A series of novel tetraphenylantimony derivatives of *exo*-7-oxa-bicyclo[2,2,1] heptane (ene)-3-arylamide-2-acid of the general formula $Ph_4SbO_2CCHCHCH_2CH_2CH(O)CHCONHAr$ (Ar = Ph,4-ClC_6H_4, 4-BrC_6H_4, 3-BrC_6H_4, 4-F-3-ClC_6H_3 and 4-MeC_6H_4) have been synthesized and characterized by elemental analysis, IR, ^1H NMR and mass spectra. The crystal structure of $Ph_4SbO_2CCHCHCH_2CH_2CH(O)CHCONHC_6H_4CH_3 \cdot CHCl_3$ was determined by X-ray diffraction. Five human neoplastic cell lines (HL-60, KB, Bel-7402, BGC-823 and HCT-8) were used to screen these compounds. The results indicate that these compounds at 10 μM show certain *in vitro* antitumor activities. Copyright © 2001 John Wiley & Sons, Ltd.

Keywords: antimony; crystal structure; antitumor activity

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INTRODUCTION

Although tetraphenylantimony carboxylates of the general formula Ph_4SbO_2CR have been known for several years, published data on the antitumor activity of these compounds is limited.[1–5]. At the same time, cantharidin (*exo*-2, 3-dimethyl-7-oxa-bicyclo[2,2,1] heptane-2,3-dicarboxylic anhydride) and its derivatives have good biological activities, e.g. both anticonvulsant[6] and antitumor activities.[7,8] In order to examine the effect of R on the structure and the antitumor activity of Ph_4SbO_2CR we have synthesized a series of tetraphenylantimony derivatives of *exo*-7-oxa-bicyclo[2,2,1] heptane (ene)-3-arylamide-2-acid, which contain two active centers, namely the tetraphenylantimony(V) moiety and the demethyl(dehydrogen) cantharidin group.

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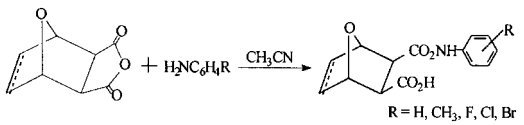
EXPERIMENTAL

General

All operations were performed in an atmosphere of dry argon using Schlenk and vacuum techniques. All solvents were dried by standard methods and distilled prior to use. Elemental analyses were determined on a Yanaco CHN Corder MT-3 elemental analyzer. IR spectra were recorded on a Bruker Equinox 55 spectrometer in KBr discs or in $CHCl_3$ solution. ^1H NMR spectra were measured on a Bruker AC-200 spectrometer in $CDCl_3$ solution with TMS as internal standard. Mass spectra (EI) were recorded on an HP-5988A at 70 eV; the ionization temperature was 200 °C.

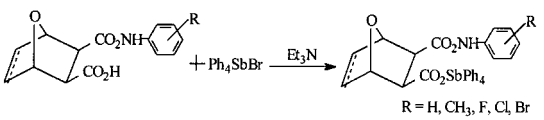
Ph_4SbBr was prepared according to the literature.[9] *exo-cis*-7-Oxa-bicyclo[2,2,1] heptane (ene)-3-arylamide-2-acid[10] was prepared by stirring a CH_3CN solution of *exo-cis*-7-oxa-bicyclo[2,2,1] heptane (ene)-2,3-dicarboxylic anhydride with primary amines in 1:1 molar ratio; the crude product was recrystallized from ethanol–petroleum

ether (Eqn [1]).



Synthesis of the title compounds

The title compounds were synthesized by a more convenient method. Recrystallized *exo-cis*-7-oxabicyclo[2,2,1] heptane (ene)-3-arylamide-2-acid (1 mmol) and triethyl amine (0.6 ml) were stirred together in toluene (25 ml) at room temperature for 0.5 h, then tetraphenylantimony bromide (1 mmol) was added. The reaction mixture was stirred overnight (Eqn [2]). A copious white precipitate appeared. After filtration the solid was washed with water. Then the solid was dried *in vacuo* to afford the title compounds. Some data on the compounds are listed in Table 1.



Double bond (CH═CH): R = H (**1**); *p*-Br (**2**); *m*-Br (**3**); *p*-Cl (**4**); *p*-CH_3 (**5**)

Single bond (CH_2—CH_2): R = 4-F-3-Cl (**6**); *p*-Br (**7**); H (**8**); *p*-CH_3 (**9**)

Crystallography

Diffraction data for compound **9** were obtained at 293 K on a Bruker Smart 1000 diffractometer (graphite-monochromatized Mo Kα radiation, $\lambda = 0.710\,73$ Å). Of the total 7912 recorded reflections, 6634 reflections ($R_{int} = 0.0171$) were used for the structure determination and refinement. The crystal class, orientation matrix and accurate unit-cell parameters were determined by standard procedures. The intensities were corrected for absorption using the SADABS program. The structure was solved by heavy atom methods and refined by a full-matrix least squares procedure based on F^2. Non-hydrogen atoms were refined with anisotropic thermal parameters.

Crystal data: $C_{39}H_{36}NO_4Sb \cdot CHCl_3$; $0.30 \times 0.25 \times 0.20\ mm^3$; triclinic; $P\bar{1}$; $a = 12.1866(11)$, $b = 13.3298(11)$, $c = 14.5481(12)$ Å; $\alpha = 67.903(2)$, $\beta = 67.128(2)$, $\gamma = 63.304°$, $V = 1886.1(3)$ Å^3; $Z = 2$; $D_c = 1.451$ Mg m^{-3}; $F(000) = 836$; $\mu = 0.985\ mm^{-1}$; $[I > 2\sigma(I)]$ $R_1 = 0.0346$, $WR_2 = 0.0864$.

Antitumor activity

The KB cell lines and Hela cell lines were obtained from the Institute of Cancer of Tianjin. Other cell lines were derived in the National Research Laboratories of Natural and Biomimetic Drugs of Peking University. All cell lines were grown in RPMI 1640 medium with 10% fetal bovine serum, in 5% CO_2 atmosphere.

The cytotoxic activity of these compounds was assayed by the MTT method.[11] The cell lines, human immature granulocyte leukemia (HL-60), human nasopharyngeal carcinoma (KB), human hepatocellular carcinoma (Bel-7402), human gastric carcinoma (BGC-823) and human colo carcinoma (HCT-8) were used for screening. Aliquots of log-phase cells were incubated for 72 h at 37 °C with four dose levels of each organoantimony complex in

Table 1 Yields and elemental analysis of the compounds

Compound	Yield (%)	m. p. (°C)	Elemental analysis: Found(calc.) (%) C	H	N	Formula for calc.
1	59	149–151	66.51(66.30)	4.54(4.68)	2.21(2.03)	$C_{38}H_{32}NO_4Sb$
2	58	163–165	59.54(59.48)	4.01(4.07)	1.69(1.83)	$C_{38}H_{31}BrNO_4Sb$
3	91	147–149	59.47(59.48)	4.10(4.07)	1.87(1.83)	$C_{38}H_{31}BrNO_4Sb$
4	64	139–141	63.13(63.14)	4.60(4.32)	1.93(1.94)	$C_{38}H_{31}ClNO_4Sb$
5	91	140–142	66.75(66.68)	4.75(4.88)	2.23(1.99)	$C_{39}H_{34}NO_4Sb$
6	68	148–150	61.31(61.44)	4.11(4.34)	2.00(1.89)	$C_{38}H_{32}FClNO_4Sb$
7	73	181–183	59.41(59.33)	4.32(4.32)	1.77(1.82)	$C_{38}H_{33}BrNO_4Sb$
8	81	190–192	66.17(66.10)	4.86(4.96)	2.09(2.03)	$C_{38}H_{34}NO_4Sb$
9	69	187–189	66.21(66.49)	5.26(5.15)	1.76(1.99)	$C_{39}H_{36}NO_4Sb$

Table 2 Important IR data of the compounds

Compound	ν(N—H)	ν(*CON*)	$\nu_{as}(CO_2)$	$\nu_{s}(CO_2)$	$\Delta\nu(CO_2)$	ν_{as}(Sb—C)
1	3250m	1686s	1601s	1359s	242	458m
2	3248m	1685m	1594s	1361s	233	457m
3	3242w	1691m	1590s	1362s	228	456m
4	3249m	1690m	1600s	1365s	235	456m
5	3251m	1690s	1597s	1364s	233	457m
6	3250m	1686s	1592s	1362s	230	457m
7	3249m	1695s	1592s	1363s	229	457s
8	3247m	1693s	1600s	1363s	237	458m
9	3248m	1685s	1592s	1361s	231	458s
9[a]	3450w	1676m	1599m	1359w	240	457w

[a] In $CHCl_3$ solution.

triplicate. 50 μl of 0.1% MTT (Sigma) was added to each well. After 4 h incubation, the culture medium was removed, and the blue formazan in the cells was dissolved with 2-propanol by vigorous shaking. The optical density of each well was measured at 570 nm wavelength. The cytotoxicity was determined by expressing the mean optical densities for drug-treated cells at each concentration as a percentage of that of untreated cells.

RESULTS AND DISCUSSION

The title compounds were prepared under mild conditions and it was not necessary to recrystallize them. These compounds are white solids with sharp melting points. They are unaffected by atmospheric moisture and can be stored for several months without decomposition. The compounds are readily soluble in CH_2Cl_2, $CHCl_3$ and DMSO, and insoluble in benzene, toluene, petroleum ether and acetone.

IR

IR spectroscopy provides a method of assessing carboxylate coordination modes from the position of, and separation ($\Delta\nu$) between, the antisymmetric and symmetric CO_2 stretching modes.[12] Selected

Table 3 ^{1}H NMR data for the compounds

Compound	OCCH	CH—O	CH═CH	CH_2—CH_2	N—H	Ar
1	2.20–2.76 (2H, m)	4.86–5.05 (2H, m)	6.20–6.25 (2H, m)		8.40 (1H, s)	7.10–7.62 (25H, m)
2	2.29–2.68 (2H, m)	4.83–4.99 (2H, m)	6.22–6.25 (2H, m)		8.39 (1H, s)	7.25–7.56 (24H, m)
3	2.25–2.72 (2H, m)	4.89–5.04 (2H, m)	6.22–6.40 (2H, m)		8.44 (1H, s)	7.08–7.65 (24H, m)
4	2.24–2.72 (2H, m)	4.89–5.04 (2H, m)	6.20–6.38 (2H, m)		8.48 (1H, s)	7.08–7.64 (24H, m)
5	2.20–2.72 (2H, m)	4.87–5.02 (2H, m)	6.24 (2H, s)		8.32 (1H, s)	7.01–7.60 (24H, m) 2.32 (*p*-CH_3, 3H, s)
6	4.85–5.54 (2H, m)	6.23–6.60 (2H, m)		2.38–3.30 (4H, m)	8.64 (1H, s)	6.90–7.72 (23H, m)
7	2.36–2.96 (2H, m)	4.60–4.67 (2H, m)		1.25–1.88 (4H, m)	8.64 (1H, s)	6.90–7.72 (24H, m)
8	2.40–2.95 (2H, m)	4.66 (2H, s)		1.20–1.90 (4H, m)	8.30 (1H, s)	7.00–7.55 (25H, m)
9	2.38–2.92 (2H, m)	4.62–4.88 (2H, m)		1.18–1.92 (4H, m)	8.28 (1H, s)	7.00–7.55 (24H, m) 2.32 (*p*-CH_3, 3H, s)

Table 4 Fragment ions observed for compound **8**

m/z	Fragment	Intensity	*m/z*	Fragment	Intensity
429	Ph_4Sb^+	16	198	$PhSb^+$	100
93	NH_2Ph^+	21	77	Ph^+	36

IR data for compounds **1–9** are summarized in Table 2. In the IR spectra of these compounds the carboxylate bands are observed in the characteristic regions: $\nu_{as}(CO_2)$ between 1601 and 1590 cm^{-1}, $\nu_s(CO_2)$ between 1365 and 1359 cm^{-1} (Table 2). On the basis of these $\Delta\nu(CO_2)$ values (242–228 cm^{-1}) these compounds can be attributed to the same class: there are no interactions between the carbonyl oxygen atoms of the carboxylate groups and the antimony atom. For compound **9** the N—H stretching frequency in $CHCl_3$ solution is shifted to 3450 cm^{-1} from 3248 cm^{-1} in solid (KBr). We interpret these features in terms of a hydrogen-bonding interaction in the crystals of the compound involving N—H and (presumably) an oxygen of the carboxylate group. In addition, the frequencies ν_{as}(Sb—C) appear between 458 and 456 cm^{-1}; this is consistent with the literature.[13,14]

^{1}H NMR

The ^{1}H NMR data of the title compounds are listed in Table 3. The chemical shifts of the double bond (CH═CH) appear between 6.20 and 6.40 ppm. The chemical shifts of the single bond (CH_2—CH_2) appear between 1.18 and 3.30 ppm. The protons of CONH show a single peak (8.28–8.64 ppm). The protons of Ph (SbPh and ArNH) show a complex multiplet (6.90–7.72 ppm).

Mass spectra

The main fragment ions observed in the mass spectra of compound **8** are listed in Table 4. The molecular ion is never observed, but the fragment ions found are in agreement with the expected structure of the compound. The ion containing antimony ($PhSb^+$) is the base peak. Decarboxylation and dephenylation from the antimony atom are the two main breakdown patterns for this compound.

Antitumor activity

The antitumor activities of these compounds are listed in Table 5. The results of the bioassay show that these compounds exhibit certain *in vitro* activities against the five tumor lines, and have a

Table 5 Antitumor activity of the compounds *in vitro*

	Inhibition ratio (%) (10 μM)				
Compound	HCT-8 cells	KB cells	HL-60 cells	BGC-823 cells	Bel-7402 cells
1	57.8	94.8	92.9	57.3	65.6
2	23.0	36.7	84.6	59.4	44.6
3	12.8	35.2	66.4	34.4	21.1
4	46.0	92.6	89.6	46.6	61.6
5	43.2	92.4	80.5	58.7	61.7
6	51.8	76.2	76.2	42.6	42.5
7	55.7	75.1	77.4	44.1	47.2
8	75.9	90.9	85.2	57.9	56.6
9	66.4	65.8	81.2	52.1	54.6
a	15.5	14.5	−0.8	−10.2	−7.9
b	18.4	17.5	17.7	−5.9	−6.3
Ph_4SbBr	–	2.0	10.7	5.1	21.7

a: *exo-cis*-7-Oxa-bicyclo[2,2,1] heptane-3-arylamide-2-acid (R = 4-F-3-Cl; see Eqn [1]).
b: *exo-cis*-7-Oxa-bicyclo[2,2,1] heptene-3-arylamide-2-acid (R = 4-Br; see Eqn [1]).

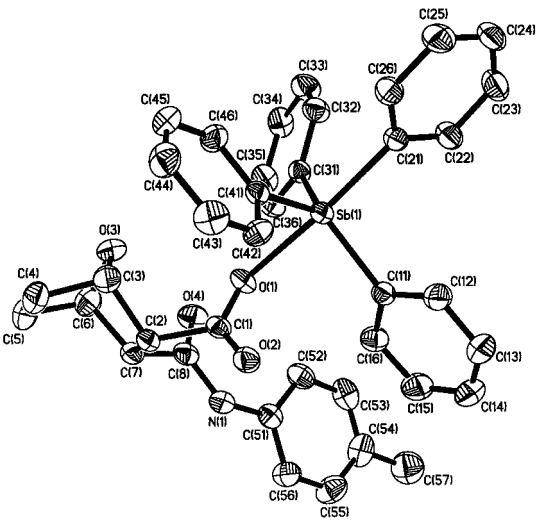


Figure 1 Molecular structure and crystallographic numbering scheme for compound **9**.

relatively higher antitumor activity than Ph_4SbBr and *exo-cis*-7-oxa-bicyclo[2,2,1] heptane (ene)-3-arylamide-2-acid (see Table 5). The results also indicate that the antitumor activities are affected by the nature of the arylamide, e.g. when R of NHC_6H_4R is a withdrawing electron group (F, Cl, Br) the compounds show relatively lower antitumor activities.

Crystal structure

A colorless single crystal of compound **9** was obtained from $CHCl_3$/petroleum ether (90–120 °C). The molecular structure of the compound is shown in Fig. 1. Selected bond lengths and angles are given in Table 6. Final fractional atomic coordinates for non-hydrogen atoms are listed in Table 7. The molecule can be considered as a distorted trigonal bipyramid with the oxygen of the carboxylate occupying an axial position. The C(21)—Sb—O(1) angle is 176.19 °. The Sb—O(1) bond length [2.278(2) Å] is close to those found in other tetraphenylantimony carboxylates (2.207–2.222 Å).[3,4] The Sb—O(2) distance is 3.488(2) Å indicating that there is no interaction between the non-bonded oxygen atom and the antimony atom, and this is consistent with the IR spectra of compound **9**. The antimony atom is displaced 0.2208 Å towards C(21) from the plane described by the equatorial carbon atoms C(11), C(31) and C(41). This leads to mean values of 96.0 ° and 84.0 ° for the angles at antimony between the equatorial carbon atoms and respectively C(21) and O(1). The distances between Sb and the carbon atoms are in the limits 2.109–2.178 Å, which is quite close to those found in pentaphenylantimony (PPA) structures.[3,4,15,16] For the 7-oxa-bicylco[2,2,1] part, the C(3)—O(3), C(6)—O(3), C(2)—C(3) and C(7)—C(8) bond lengths are 1.418(6) Å, 1.443(6) Å, 1.549(5) Å and 1.528(5) Å respectively, whereas

Table 6 Selected bond distances and bond angles of compound **9**

Bond	Distances (Å)	Bond	Angles (°)
Sb(1)—C(41)	2.107(3)	C(41)—Sb(1)—C(11)	121.41(12)
Sb(1)—C(11)	2.110(3)	C(41)—Sb(1)—C(31)	117.47(12)
Sb(1)—C(31)	2.130(3)	C(11)—Sb(1)—C(31)	117.89(13)
Sb(1)—C(21)	2.178(3)	C(41)—Sb(1)—C(21)	96.33(12)
Sb(1)—O(1)	2.278(2)	C(11)—Sb(1)—C(21)	96.47(13)
Sb(1)—O(2)	3.488(2)	C(31)—Sb(1)—C(21)	95.14(12)
C(2)—C(3)	1.549(5)	C(41)—Sb(1)—O(1)	82.63(10)
C(2)—C(7)	1.558(5)	C(11)—Sb(1)—O(1)	87.18(11)
C(3)—C(4)	1.534(6)	C(31)—Sb(1)—O(1)	82.15(10)
C(4)—C(5)	1.522(8)	C(21)—Sb(1)—O(1)	176.19(10)
N(1)—O(2)	2.850	C(1)—O(1)—Sb(1)	125.8(2)
O(1)—C(1)	1.276(4)	O(2)—C(1)—O(1)	125.1(3)
O(2)—C(1)	1.228(4)	O(1)—C(1)—C(2)	115.6(3)
O(3)—C(3)	1.418(6)	C(3)—O(3)—C(6)	96.1(3)
O(3)—C(6)	1.443(6)	O(2)—C(1)—C(2)	119.2(3)
O(4)—C(8)	1.216(4)	N(1)—H—O(2)	160.81
C(5)—C(6)	1.518(7)		
C(6)—C(7)	1.524(5)		

Table 7 Fractional coordinates and thermal parameters of non-hydrogen atoms for compound **9**

	x	*y*	*z*	U_{eq}
Sb(1)	7202(1)	8556(1)	6485(1)	39(1)
C(9)	6194(5)	7032(5)	10367(4)	91(2)
Cl(1)	5847(2)	6345(2)	9789(2)	161(1)
Cl(2)	7200(2)	6042(1)	11151(1)	100(1)
Cl(3)	4849(2)	7892(3)	11018(3)	212(2)
N(1)	10503(3)	4216(2)	6822(2)	50(1)
O(1)	9097(2)	7099(2)	6113(2)	47(1)
O(2)	8833(2)	6565(2)	4962(2)	54(1)
O(3)	11724(3)	7155(3)	5450(3)	89(1)
O(4)	11132(3)	5136(2)	7451(2)	64(1)
C(1)	9507(3)	6642(3)	5359(2)	42(1)
C(2)	10954(3)	6233(3)	4894(3)	45(1)
C(3)	11440(4)	7243(4)	4559(4)	73(1)
C(4)	12754(5)	6963(4)	3783(4)	88(2)
C(5)	13583(4)	6059(5)	4491(5)	91(2)
C(6)	12618(4)	5980(4)	5536(4)	76(1)
C(7)	11814(3)	5329(3)	5617(3)	50(1)
C(8)	11107(3)	4908(3)	6726(3)	49(1)
C(11)	6313(3)	7373(3)	6816(3)	43(1)
C(12)	5140(4)	7779(3)	6632(3)	59(1)
C(13)	4521(4)	7017(4)	6888(4)	80(1)
C(14)	5048(5)	5874(4)	7352(4)	83(1)
C(15)	6204(5)	5476(4)	7545(4)	77(1)
C(16)	6852(4)	6217(3)	7265(3)	59(1)
C(21)	5458(3)	10010(3)	6847(3)	45(1)
C(22)	4603(4)	9858(3)	7804(3)	58(1)
C(23)	3534(4)	10789(4)	8087(4)	70(1)
C(24)	3299(4)	11859(4)	7430(4)	74(1)
C(25)	4128(4)	12006(3)	6474(4)	76(1)
C(26)	5217(4)	11088(3)	6176(3)	59(1)
C(31)	7957(3)	8452(3)	7628(2)	41(1)
C(32)	7468(4)	9397(3)	8040(3)	58(1)
C(33)	7894(4)	9326(4)	8830(3)	69(1)
C(34)	8796(4)	8335(4)	9200(3)	66(1)
C(35)	9287(4)	7416(4)	8782(3)	61(1)
C(36)	8890(3)	7460(3)	7992(3)	52(1)
C(41)	7899(3)	9407(3)	4960(2)	39(1)
C(42)	7693(3)	9282(3)	4147(3)	49(1)
C(43)	8136(4)	9865(4)	3155(3)	65(1)
C(44)	8775(4)	10571(4)	2984(3)	66(1)
C(45)	8976(4)	10714(4)	3772(3)	63(1)
C(46)	8522(4)	10132(3)	4777(3)	54(1)
C(51)	9820(3)	3647(3)	7737(3)	49(1)
C(52)	9270(4)	3992(4)	8648(3)	62(1)
C(53)	8585(4)	3390(4)	9498(3)	72(1)
C(54)	8454(4)	2429(5)	9469(3)	74(1)
C(55)	9015(5)	2097(4)	8559(4)	82(1)
C(56)	9677(4)	2690(4)	7712(3)	65(1)
C(57)	7718(6)	1770(5)	10403(5)	108(2)

in cantharidin the corresponding C—O and C—C distances are 1.447 Å and 1.558 Å.[17]

Inspection of the molecular packing of the compound shows that two molecules are held together forming a molecular pair in the lattice by two relatively strong hydrogen-bonding interac-

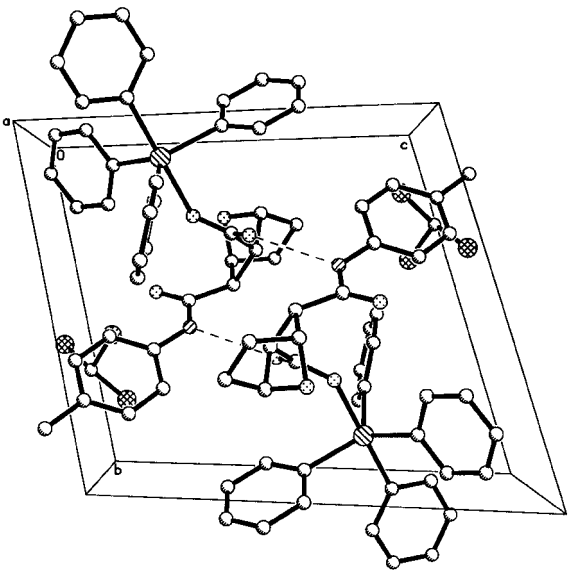


Figure 2 Unit cell for compound **9** showing hydrogen-bonding interactions.

tions between the amide hydrogen and the carboxylate carbonyl oxygen of an adjacent molecule. The N(1)—O(2) distance is 2.850 Å, which is less than the sum of the van der Waals radii of 3.11 Å.[18] The participation of the carboxyl oxygen in hydrogen bonding clearly makes interaction between Sb and O(2) impossible.

SUPPLEMENTARY MATERIAL

Crystallographic data for the structure analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC no. 149419. Copies of this information may be obtained free of charge from: The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK; fax: +44-(1223)-336033; email: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk.

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