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Triorganoantimony(V) carboxylates: Synthesis, characterization and crystal structure of $[Me_3Sb(O_2C–C_5H_4N)_2]\cdot H_2O$

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Abstract

Reactions of $[R_3Sb(OPr^i)_2]$ with *N*-heterocylic carboxylic acids gave compounds of the type $[R_3Sb(O_2C–Ar)_2]$ (**1**) (R = Me, Et, Pr^i, Ph; Ar = 2-C_5H_4N, 2-C_9H_6N). The mono-bromo compound $[Me_3Sb(Br)(O_2C–C_5H_4N)]$ (**2**) exists in equilibrium with $[Me_3Sb(O_2C–C_5H_4N)_2]$ and $[Me_3SbBr_2]$. All new compounds have been characterized by IR and NMR (^1H and $^{13}C\{^1H\}$) spectral data. X-ray structural analysis of one example, $[Me_3Sb(O_2C–C_5H_4N)_2]$, isolated as its monohydrate, revealed an essentially trigonal bipyramidal geometry for the antimony atom defined by three equilaterally disposed methyl groups and two oxygen atoms from monodentate carboxylate groups, in apical positions. The crystal structure is consolidated into a three-dimensional network by cooperative O–H···O, O–H···N and C–H···O interactions.

Keywords: Triorganoantimony; Picolinic acid; Quinaldic acid; NMR; X-ray structure

1. Introduction

Organoantimony(V) complexes containing an Sb–O–R linkage (where OR = alkoxy, phenoxy, carboxylate, oximate) have been investigated in considerable detail [1–5]. Their mono- and di-organoantimony(V) complexes are quite often dimeric [1,6] whereas tri- and tetra-organoantimony derivatives are monomeric, with the central metal atom acquiring a trigonal bipyramidal configuration. Anionic bidentate ligands, such as acetylacetonate$^-$, oxinate$^-$ (8-hydroxyquinolate ion), Schiff bases, *etc*., in general, yield hexa-coordinated tri- and tetra-organoantimony complexes [1,7,8].

2-Picolinic acid and related carboxylic acids have been used to stabilize higher coordination either through O^N chelation or via carboxylate bridges [9–11]. To assess their coordination behaviour towards triorganoantimony(V) moieties, a series of compounds have been synthesized and one of them, namely $[Me_3Sb(O_2C–C_5H_4N)_2]$, has been fully characterized by single-crystal X-ray crystallography as its monohydrate. Results of this work are described herein.

2. Results and discussion

2.1. Synthesis and spectroscopic characterization

Reactions of $[R_3Sb(OPr^i)_2]$ with two equivalents of *N*-heterocyclic carboxylic acids in benzene afforded colourless bis(carboxylates), $[R_3Sb(O_2C–Ar)_2]$ (R = Me, Et, Pr^i, Ph; Ar = C_5H_4N, C_9H_6N) in nearly quantitative yields as per Eq. (1). The IR spectra displayed absorptions in the region 1678–1630 cm^{-1} attributable to ν(C=O) indicating the presence of a free carbonyl group. The ν(Sb–C) absorptions in trialkylantimony(V) compounds have been assigned in the region 495–565 cm^{-1} [12]. The ^1H and $^{13}C\{^1H\}$ NMR spectra displayed characteristic peaks assignable to $R_3Sb(V)$ and the carboxylate fragments.

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The resonances of the $R_3Sb(V)$ fragment due to the α-carbon and the protons attached to this carbon are considerably shielded from the corresponding signals for the corresponding dibromide (R_3SbBr_2) in the $^{13}C\{^1H\}$ and ^1H NMR spectra, respectively. This shielding can be ascribed to the coordination of more electronegative oxygen, compared with bromide, to the antimony atom. Nitrogen coordination of heterocyclic aprotic ligands to metal atoms is manifested in the form of deshielding of the C-4 carbon resonance by ~3 ppm [7,9,10,13]. In the present case, the C-4 resonance of 2-picolinate is little affected on coordination of the carboxylate ligands from its position for the free ligand suggesting an absence of Sb–N coordination, which was confirmed by X-ray crystal structure of **1a** (see below).

$$[R_3Sb(OPr^i)_2] + 2ArCO_2H \rightarrow [R_3Sb(O_2C\text{–}Ar)_2] + 2Pr^iOH \quad (R = Me, Et, Pr^i, Ph; Ar = 2\text{-}C_5H_4N, 2\text{-}C_9H_6N) \qquad (1)$$

Attempts to prepare $[Me_3Sb(Br)(O_2C\text{–}C_5H_4N)]$ by the reaction of Me_3SbBr_2 with one equivalent of $NaO_2C\text{–}C_5H_4N$ lead to the formation of a mixture containing Me_3SbBr_2, $[Me_3Sb(Br)(O_2C\text{–}C_5H_4N)]$, (**2**) and (**1a**), as revealed by NMR spectroscopy. A similar equilibrium was established when $CDCl_3$ solutions of **1a** and $[Me_3SbBr_2]$ were mixed in 1:1 stoichiometry at room temperature as per Eq. (2). The relative ratio did not change even after refluxing the solution for 3 h. The ^1H NMR spectrum in C_6D_6 also showed the presence of these three species; only the chemical shifts showed the expected solvent effect. However, ^1H NMR spectra recorded in DMSO-d_6 and CD_3OD displayed a broad signal for $Me_3Sb(V)$ protons, suggesting the exchange process faster on NMR time scale in these solvents.

$$[Me_3Sb(O_2C\text{–}C_5H_4N)_2] + Me_3SbBr_2 \rightleftharpoons 2[Me_3Sb(Br)(O_2C\text{–}C_5H_4N)] \qquad (2)$$

Anionic bidentate ligands, such as acetylacetonate$^-$, oxinate$^-$, readily form six-coordinated $[R_3SbX(L)]$ (X = Cl or Br; L = acetylacetonate, oxinate) containing chelating ligands. These complexes are stable both in solution and in the solid-state. Surprisingly, compound **2** exists in equilibrium with **1a** and Me_3SbBr_2 in solution, although 2-picolinate ion is known to give five-membered O^N chelated metal complexes similar to oxinate [1,10].

2.2. Crystallographic structure of $[Me_3Sb(O_2C\text{–}C_5H_4N)_2]\cdot H_2O$

The molecular structure of $[Me_3Sb(O_2C\text{–}C_5H_4N)_2]$, characterised as its monohydrate, is illustrated in Fig. 1 which shows the hydrogen-bonds formed between the components of the crystallographic asymmetric unit; selected geometric parameters are listed in the caption to the figure. The antimony atom exists within a *trans*-C_3O_2 donor set defined by three methyl-carbon atoms and two oxygen atoms, derived from two essentially monodentate carboxylate ligands; the axial O1–Sb–O3 is 171.91(15)°. The coordination geometry is based on a trigonal bipyramid but there are significant distortions from the ideal geometry. As can be seen from Fig. 1, the carboxylate ligands adopt different orientations with respect to the central antimony atom. The O3,O4-carboxylate adopts the conventional orientation in which the O4 atom is directed towards the antimony atom, being separated by 3.024(4) Å. The O1,O2-carboxylate ligand adopts a different orientation so as to place the pyridine-N1 in close proximity to the antimony atom; the Sb···N1 distance is 2.665(5) Å. While neither distance is indicative of a significant bonding interaction to the antimony atom, the close approach of these atoms is responsible for the widening of the trigonal C13–Sb–C14 angle to 146.3(2)° compared with the narrower C13–Sb–C15 and C14–Sb–C15 angles of 105.5(2)° and 107.8(2)°, respectively. Each of the two 2-picolinate ligands is approximately planar as manifested in the O1–C6–C1–N1 and O3–C12–C7–N2 torsion angles of 6.8(7)° and −17.8(8)°, respectively. Further, the dihedral angle between the pyridine rings is only 23.5(3)°, indicating that the molecule almost achieves mirror symmetry. As mentioned above, the water molecule is associated with the $[Me_3Sb(O_2C\text{–}C_5H_4N)_2]$ molecule and from Fig. 1 it is clear that it straddles the O3 and N2 atoms. The water molecule is somewhat

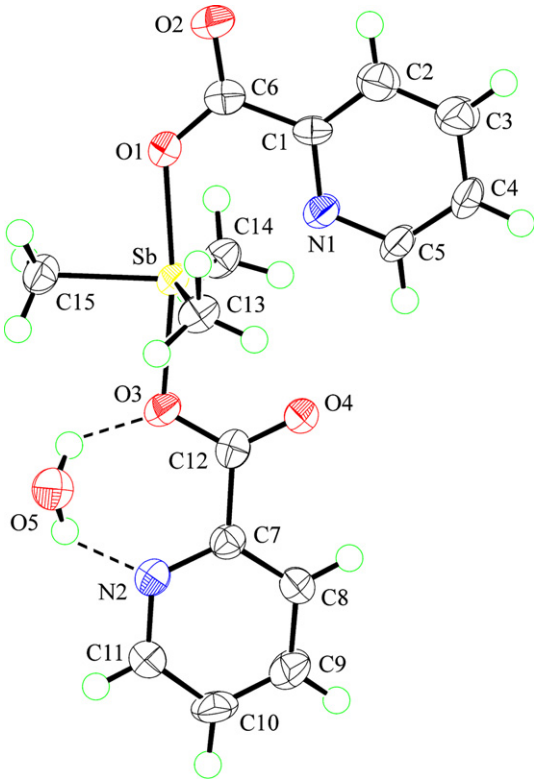

Fig. 1. Molecular structure and crystallographic labelling scheme for the crystallographic asymmetric unit of $[Me_3Sb(O_2C\text{–}C_5H_4N)_2]\cdot H_2O$. The hydrogen-bonds formed between the water molecule and $[Me_3Sb(O_2C\text{–}C_5H_4N)_2]$ are shown as black-dashed lines. Selected geometric parameters: Sb–O1 2.135(4), Sb–O3 2.154(4), Sb–C13 2.093(5), Sb–C14 2.091(6), Sb–C15 2.117(6), O1–C6 1.300(7), O2–C6 1.231(7), O3–C12 1.295(7), O4–C12 1.230(7) Å; O1–Sb–O3 171.91(15)°.

removed from the O3–C12–C7–N2 plane and forms rather long O–H···O, N separations [14]. It is easy to envisage the water molecule moving in closer to the basic atoms. However, its relative position in the crystal structure allows for the relatively close approach of adjacent molecules and hence, the formation of C–H···O contacts. Indeed, the water-O5 atom accepts two such contacts [14] from two symmetry related molecules and thereby plays a pivotal role in the stabilisation of the crystal structure. Additional C–H···O contacts involve the carboxylate-O2 and -O4 atoms [14]. A view of the crystal packing diagram is shown in Fig. 2 which illustrates the cohesiveness of the crystal structure owing to the presence of the various intermolecular interactions, detailed above, that extend in three-dimensions.

A survey of the Cambridge Crystallographic Database [15] indicated that there are 32 triorganoantimony dicarboxylate structures of which only four had antimony-bound methyl substituents [16,17], the remaining having aromatic groups bound to antimony. Of the trimethylantimony structures, arguably the most relevant for comparison is that of $[Me_3Sb(O_2CCH_2–C_5H_4N\text{-}2)]$, i.e. with a $–CH_2–$ bridge between the carboxylato and pyridine residues [17]. Here, not surprisingly, both oxygen atoms of each carboxylato residue of each of the two crystallographically independent molecules are oriented towards the antimony atom as the positions of the nitrogen atoms preclude intramolecular association to the central atom [17]. In this structure, the range of $Sb–O_{short}$ distances is 2.119(3)–2.133(3) Å, and $Sb–O_{long}$ distance range is 3.012(6)–3.112(4) Å, i.e. akin to those seen in the structure of $[Me_3Sb(O_2C–C_5H_4N)_2]\cdot H_2O$. Of the triarylantimony dicarboxylates, the most relevant structure of comparison is that of $[Ph_3Sb(O_2C–C_5H_4N)_2]$ [17]. Here, the presence of the somewhat electronegative phenyl groups increases the Lewis acidity of the antimony atom. While the orientation of the 2-picolinate ligands matches those found in $[Me_3Sb(O_2C–C_5H_4N)_2]\cdot H_2O$, the non-bonding atoms approach the antimony atom at closer distances. Thus, for the carboxylate ligand with both oxygen atoms directed towards the antimony atom, the Sb–O distances are 2.185(2) Å and 2.721(3) Å. The other carboxylate ligand

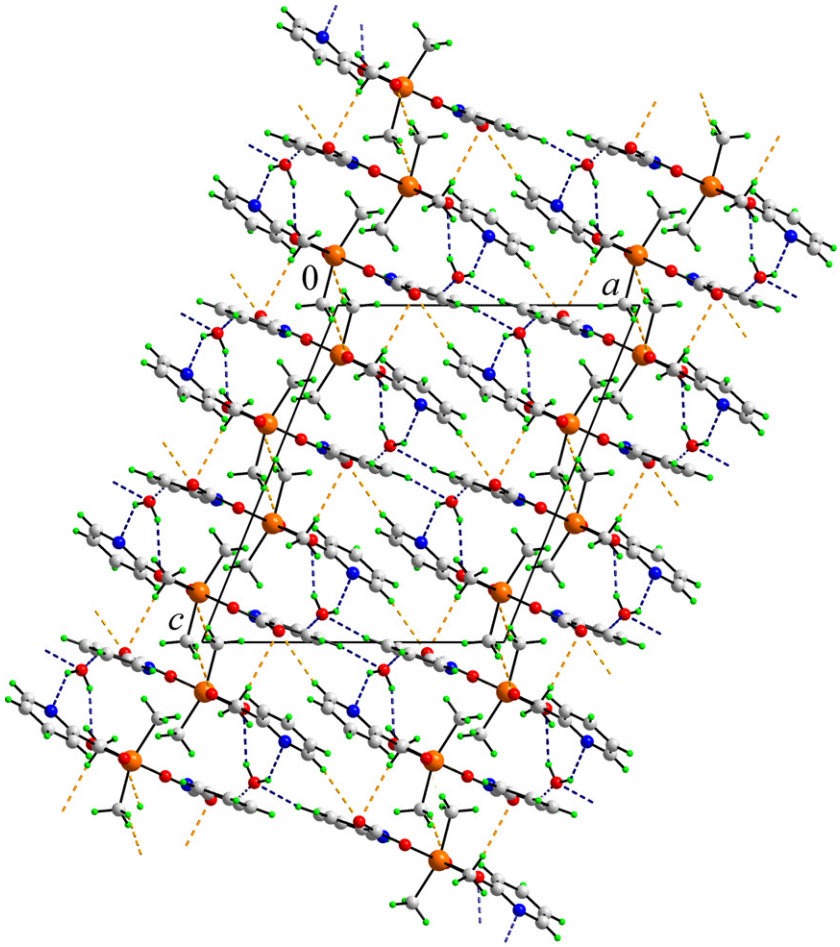

Fig. 2. Crystal packing diagram for $[Me_3Sb(O_2C–C_5H_4N)_2]\cdot H_2O$ viewed down the *b*-axis. The hydrogen-bonds involving the lattice water molecule are shown as blue-dashed lines. The C–H···O contacts involving the carboxylate-O2, O4 atoms are shown as orange-dashed lines. Colour code: antimony: orange; oxygen: red; nitrogen: blue; carbon: grey; hydrogen: green. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

forms Sb–O, N separations of 2.099(2) Å and 2.600(3) Å [17]. If the donor atoms forming the longer distances in $[Ph_3Sb(O_2C–C_5H_4N)_2]$ are considered to be bonding, the coordination geometry would be best described as being based on a pentagonal bipyramid with phenyl groups occupying axial positions.

3. Experimental

3.1. Reagents and instrumentation

All preparations involving organoantimony compounds were performed in Schlenk flask in anhydrous condition under a nitrogen atmosphere. Antimony trichloride, 2-picolinic acid and 2-quinaldic acid were obtained from S.D. Fine Chemicals. Triorganostibines, R_3Sb (R = Me, Et, Pr^i, Ph), were obtained by the reaction of $SbCl_3$ with RMgX (X = Br or I) in diethylether and their oxidation by a CCl_4 solution of bromine gave corresponding R_3SbBr_2. Triorganoantimony(V) isopropoxides, $[R_3Sb(O\text{-}Pr^i)_2]$, were prepared by the reaction of R_3SbBr_2 with NaO-Pr^i in isopropanol–benzene and the trialkyl derivatives were distilled under reduced pressure and their purity was ascertained by ^{1}H NMR spectra [18]. Infrared spectra were recorded between CsI plates on a Bomen MB-102 FT IR spectrophotometer. NMR spectra (^{1}H and $^{13}C\{^1H\}$) were recorded on a Bruker DPX-300 spectrometer in 5 mm thin walled NMR tube as $CDCl_3$ solutions. Chemical shifts are relative to internal chloroform peak (7.26 ppm and 77.0 ppm for ^{1}H and $^{13}C\{^1H\}$NMR, respectively).

3.2. Synthesis

3.2.1. $[Me_3Sb(O_2C–C_5H_4N)_2]$ (**1a**)

To a benzene solution (60 cm^3) of $[Me_3Sb(OPr^i)_2]$ (752 mg, 2.64 mmol) was added 2-picolinic acid (649 mg, 5.28 mmol) under a nitrogen atmosphere and the whole was stirred at room temperature for 3 h. The solvent was evaporated under vacuum to give a colourless solid (1.023 g, 94%). This was recrystallized from benzene–hexane mixture, m.p. 136 °C. Anal. Calc. for $C_{15}H_{17}N_2O_4Sb$: C, 43.8; H, 4.2; N, 6.8. Found: C, 43.2; H, 4.9; N, 6.7%. IR in Nujol: 1654 (*v* CO), 565 (*v* Sb–C) cm^{-1}. ^{1}H NMR in $CDCl_3$: 2.09 (s, Sb*Me*$_3$); 7.44 (m), 7.82 (m), 8.12 (d, 7.7 Hz); 8.82 (br) (C_5H_4N). $^{13}C\{^1H\}$NMR in $CDCl_3$: 14.1(s, Sb*Me*$_3$); 125.0 (C–5), 126.0 (C-3), 136.8 (s) (C-4), 149.2 (s) (C-6), 150.3 (C-2); 169.0 (s, CO). All other complexes were prepared similarly by the reaction between $R_3Sb(OPr^i)_2$ and heterocyclic carboxylic acid.

3.2.2. $[Me_3Sb(O_2C–C_9H_6N)_2]$ (**1b**)

98% yield, m.p 121 °C. IR in Nujol: 1630 cm^{-1} *v*(C=O). ^{1}H NMR in $CDCl_3$: 2.25 (s, *Me*$_3$Sb); 7.63 (t, 7 Hz, H-5), 7.79 (t, 7 Hz, H-8), 7.87 (d, 8.1 Hz, H-4); 8.24 (AB pattern, H-6, H-7); 8.40 (d, 8.5 Hz, H-3). $^{13}C\{^1H\}$NMR in $CDCl_3$: 13.2 (s, *Me*$_3$*Sb*); 121.4, 127.4, 128.3,129.2, 130.0, 130.9, 137.0, 147.7, 150.5; 169.2 (CO).

3.2.3. $[Et_3Sb(O_2C–C_5H_4N)_2]$ (**1c**)

84% as a paste. IR in Nujol: 1650 *v*(CO), 550 *v*(Sb–C) cm^{-1}. ^{1}H NMR in $CDCl_3$: 1.42 (t, 8 Hz, $SbCH_2CH_3$); 2.54 (q, 8 Hz, $SbCH_2$); 7.34 (t, 8 Hz), 7.73 (t, 8 Hz), 8.04 (d, d, 0.8, 8.0 Hz), 8.76 (br) $C_5H_4N^-$. $^{13}C\{^1H\}$NMR in $CDCl_3$: 9.0 (s, $SbCH_2CH_3$); 25.2 (s, $SbCH_2$); 124.4 (C-5), 125.6 (C-3), 136.5 (C-4), 149.0 (C-6), 150.2 (C-2), 168.6 (s, C=O).

3.2.4. $[Et_3Sb(O_2C–C_9H_6N)_2]$ (**1d**)

87% yield as paste. IR in Nujol: 1647 *v* (C=O), 495 *v* (Sb–C) cm^{-1}. ^{1}H NMR in $CDCl_3$: 1.61 (t, 7.8 Hz, $SbCH_2CH_3$); 2.79 (q, 7.8 Hz, $SbCH_2$); 7.60 (t, 7 Hz, H-5), 7.78 (t, 7 Hz, H-8), 7.85 (d, 8.1 Hz, H-4); 8.20 (AB quartet, H-6, 7); 8.45 (d, 8.5 Hz, H-3). $^{13}C\{^1H\}$NMR in $CDCl_3$: 9.2 (s, $SbCH_2CH_3$); 24.6 (s, $SbCH_2$); 121.1, 127.1, 127.7, 128.8, 129.7, 130.5, 166.6, 147.5, 150.5; 168.8 (C=O).

3.2.5. $[Pr^i_3Sb(O_2C–C_5H_4N)_2]$ (**1e**)

87% yield. IR in Nujol: 1650 *v*(C=O), 557 *v*(Sb–C) cm^{-1}. ^{1}H NMR in $CDCl_3$: 1.68 (d, 7.1 Hz, SbCH*Me*$_2$); 3.39 (sep, 7.1 Hz, SbCH-); 7.39 (br), 7.79 (br), 8.04 (d, 7.6 Hz), 8.74 (br). $^{13}C\{^1H\}$NMR in $CDCl_3$: 19.7 (s, SbCH*Me*$_2$); 39.9 (s, SbCH<); 124.1(C-5), 125.2 (C-3), 136.0 (C-4), 149.4 (C-6), 150.8 (C-2), 168.9 (C=O).

3.2.6. $[Pr^i_3Sb(O_2C–C_9H_6N)_2]$ (**1f**)

94% yield, m.p. 128 °C. Anal. Calc. for $C_{29}H_{33}N_2O_4Sb$: C, 58.5; H, 5.6; N, 4.7. Found: C, 58.3; H, 5.3; N, 5.3%. IR in Nujol: 1640 *v*(C=O), 538 *v*(Sb–C) cm^{-1}. ^{1}H NMR in $CDCl_3$: 1.78 (d, 7.1 Hz, SbCH*Me*$_2$); 3.51 (sep, 7.1 Hz, Sb*CH*<); 7.60 (t, 7 Hz, H-5); 7.75 (t, 7 Hz, H-8); 7.85 (d, 7.5 Hz, H-4); 8.20 (AB pattern, H-6, 7), 8.35 (d, 8.3 Hz, H-3). $^{13}C\{^1H\}$NMR in $CDCl_3$: 20.2 (s, SbCH*Me*$_2$); 40.4 (s, Sb*CH*<); 121.3; 127.3, 127.8, 129.0, 129.7, 131.1, 136.7, 148.1, 151.2, (C_9H_6N), 169.3 (C=O).

3.2.7. $[Ph_3Sb(O_2C–C_5H_4N)_2]$ (**1g**)

Yield 85%, m.p. 120 °C. IR in Nujol: 1678 cm^{-1}*v*(C=O). ^{1}H NMR in $CDCl_3$: 7.29–7.31 (m, C_6H_5Sb); 7.48 (t, 7 Hz, C_5H_4N); 7.79–7.88 (m, $C_6H_5 + C_5H_4N$); 8.14 (d, 7.8 Hz, C_5H_4N); 9.22 (d, 5 Hz, C_5H_4N). $^{13}C\{^1H\}$NMR in $CDCl_3$: 125.4 (C-5), 126.6 (C-3), 129.1 (C-3, 5, Ph), 130.3 (C-4, Ph), 133.3 (C-2,6; Ph), 133.8 (Sb–C), 137.7 (C-4), 145.4 (C-6), 148.8 (C-2), 167.7 (C=O).

3.2.8. $[Ph_3Sb(O_2C–C_9H_6N)_2]$ (**1h**)

Yield 97% m.p 131 °C. IR in Nujol: 1645 cm^{-1} *v*(CO). ^{1}H NMR in $CDCl_3$: 7.15– 8.82 (m, Ph + C_9H_6N).

3.2.9. $[Me_3Sb(Br)(O_2C–C_5H_4N)]$ (**2**)

Reaction between Me_3SbBr_2(136 mg, 0.42 mmol) and $[Me_3Sb(O_2C–C_5H_4N)_2]$ (172 mg, 0.42 mmol) in benzene gave a mixture containing $[Me_3Sb(O_2C–C_5H_4N)_2]$, $[Me_3Sb(Br)(O_2C–C_5H_4N)]$ and Me_3SbBr_2 with a relative ratio of 1:2:1. This ratio did not change even after refluxing the solution for 3 h. The trimethylantimony ^{1}H NMR signals for this product in different solvents are given below:

$CDCl_3$: 2.06 [$Me_3Sb(O_2C–C_5H_4N)_2$],
2.37 [$Me_3Sb(Br)(O_2C–C_5H_4N)$]
2.61 [Me_3SbBr_2]
C_6D_6: 1.80 [$Me_3Sb(O_2C–C_5H_4N)_2$]
1.98 [$Me_3Sb(Br)(O_2C–C_5H_4N)$]
2.05 [Me_3SbBr_2]
DMSO-d_6: only one broad resonance δ: 2.07.
CD_3OD: only one broad resonance δ: 2.25 ($1/2\Delta = 36$ Hz).

3.3. Crystal structure determination

Crystals of $[Me_3Sb(O_2C–C_5H_4N)_2]\cdot H_2O$ were obtained from the slow evaporation of a dichloromethane/hexane solution of the compound (**1a**). Intensity data for a colourless crystal $0.03 \times 0.24 \times 0.32$ mm^3 were collected at 120 K on a Bruker SMART APEX2 CCD using Mo Kα radiation so that $\theta_{max} = 27.5°$. The data set was corrected for absorption based on multiple scans [19] and reduced using standard methods [20]. The structure was solved by heavy-atom methods [21] and refined by a full-matrix least-squares procedure on F^2 with anisotropic displacement parameters for non-hydrogen atoms, carbon-bound hydrogen atoms in their calculated positions and a weighting scheme of the form $w = 1/[\sigma^2(F_0^2) + (0.060P)^2 + 1.112P]$ where $P = (F_0^2 + 2F_c^2/3)$ [22]. The water–hydrogen atoms were located from a difference map and refined with O–H constrained to 0.840(1) Å. Fig. 1, showing the atom labelling scheme, was drawn with 70% displacement ellipsoids using ORTEP [23] and Fig. 2 was drawn with DIAMOND with arbitrary spheres [24]. Data manipulation and interpretation were accomplished using teXsan [25] and PLATON [26]. Crystal data for $[Me_3Sb(O_2C–C_5H_4N)_2]$: $C_{15}H_{19}N_2O_5Sb$, $M = 429.07$, monoclinic, $P2_1/c$, $a = 12.2264(9)$ Å, $b = 10.1651(7)$ Å, $c = 14.6901(8)$ Å, $\beta = 111.959(4)°$, $V = 1693.27(19)$ Å^3, $Z = 4$, $D_x = 1.683$ g cm^{-3}, $\mu = 1.655$ mm^{-1}, R (2580 data with $I \geqslant 2\sigma(I)) = 0.040$, wR = (all 3874 data) = 0.126.

Acknowledgements

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Appendix A. Supplementary material

CCDC 649446 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2007.07.033.

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