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# Triphenylantimony(V) derivatives of acylpyrazol-5-ones

M.F. Mahon [a], K.C. Molloy [a,*], B.A. Omotowa [1,a], M.A. Mesubi [b]

[a] *School of Chemistry, University of Bath, Bath BA2 7AY, UK*
[b] *Department of Chemistry, University of Ilorin, P.M.B. 1515 Ilorin, Nigeria*

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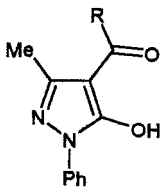
**Abstract**

The synthesis and characterisation of two bis(triphenylantimony)oxo-4-acylpyrazol-5-ones (4-Me, 4-Ph) and one example of an analogous derivative of the isomeric ligand 1-acetylpyrazol-5-one are reported. The structure of one compound, $[Ph_3Sb(L)]_2O$ (L = 1-phenyl-3-methyl-4-benzoylpyrazol-5-one), has been determined; each antimony is in a six-coordinated $Ph_3SbO_3$ environment with a *mer*-arrangement of the ligand sets. 

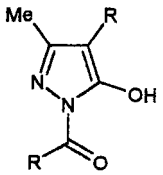
*Keywords:* Triphenylantimony; Acylpyrazol-5-one derivatives; Antimony; X-ray

## 1. Introduction

4-Acyl-5-pyrazolones (I) are a widely explored ligand class [1], but one whose organometallic chemistry is only currently being discovered.




(I)




(II)

Rhodium and iridium olefine complexes were the earliest receptors for these ligands [2], while the later work of Saxena and, primarily, Pettarinari on diorganotin derivatives has been more extensive and therefore more significant [3–8]. We have contributed the synthesis and structural characterisation of the first triorganotin complexes of these ligands (also reported subsequently by Pettarinari [9]) along with tri- and di-organotin derivatives of isomeric 1-acylpyrazol-5-ones (II) [10] and, more recently, organomercury (II) and diorganothallium(III) derivatives of (I) [11]. The mercury complex is significant in that it represents the first crystallographically authenticated oxygen-bonded organomercury $\beta$-diketonate [11]. We now wish to report the first organoantimony derivatives of this ligand class, along with the structural characterisation of $\mu$-oxo-bis[triphenyl(1-phenyl-3-methyl-4-benzoylpyrazol-5-onato)antimony (V)] (**1**).

* Corresponding author.
[1] On leave from the University of Ilorin.

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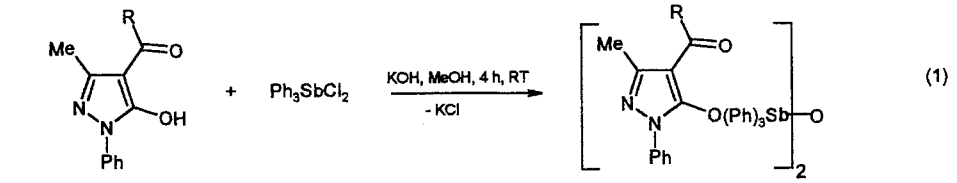
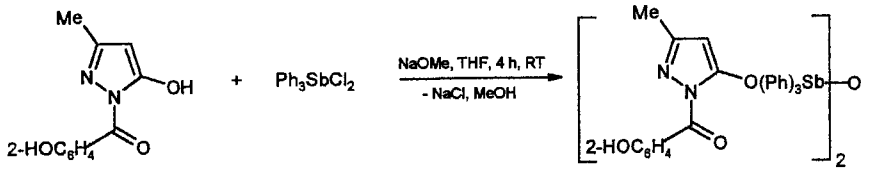
## 2. Results and discussion

### 2.1. Synthesis and spectroscopy




(1)

R = Me (1), Ph (2)




(2)

(3)

Three new $\mu$-oxo-bis[(acylpyrazol-5-onato)triphenylantimony(V)] complexes of the type $(Ph_3SbL)_2O$ have been successfully synthesised by stirring either 1:1 or 1:2 molar equivalents of triphenylantimony(V) dichloride and the ligand in a methanolic solution containing 1 or 2 equivalents of potassium hydroxide for 4 h at r.t. Two of these compounds (**1**, **2**) are derivatives of 4-acylpyrazol-5-one (Eq. 1) while the third (**3**) is bonded to the isomeric 1-acylpyrazol-5-one (Eq. 2). In this latter case, NaOMe rather than KOH was used as base.

Interestingly, $\beta$-diketonate complexes $(R_3SbL)_2O$ (R = Ph, 4-$ClC_6H_4$; L = $\beta$-diketone) have previously been prepared from $R_3SbBr_2$, $\beta$-diketone and $Et_3N$ in dry benzene [12]. In addition, several compounds of the type $(Me_3SbL)_2O$ [L = $S_2CNR$, $S_2COR$, $S_2P(OR)_2$] have been synthesised from the reaction of $\mu$-oxo-bis(bromotrimethylantimony), $(Me_3SbBr)_2O$, with the sodium salt of the ligand in dichloromethane [13].

The new oxo-bridged complexes are white crystalline (**1**, **2**) or pale yellow (**3**), high melting solids which are soluble in methanol, chloroform, THF and benzene, but insoluble in hexane and water. They were recrystallised from toluene/ether at r.t. without decomposition and are reasonably stable in air, but should be kept in a vacuum desiccator for long storage.

The synthesis and chemical properties of a number of compounds of the type $R_3Sb(X)L$, [R = Ph, 4-$MeC_6H_4$; X = halogen; LH = acetylacetone, ethyl acetoacetate, diethyl malonate] have been reported recently, as have cationic triorganoantimony(V) $\beta$-diketonates e.g. $[R_3Sb(acac)][ClO_4]$ (R = Ph or 4-$MeC_6H_4$). Interestingly, when the dihalides, $R_3SbX_2$ (X = Br or Cl) were refluxed with the sodium salt of the $\beta$-diketone or $\beta$-diketo ester in benzene solution, only one halogen was displaced to give the compounds $R_3Sb(X)L$, irrespective of whether one or 2 molar equivalents of the sodium salt of the ligand were used. Compounds of the type $R_3Sb(L')L$ (L = $O_2CMe$ or OMe) were prepared from $R_3Sb(X)L$ and sodium acetate or sodium methoxide, respectively and were reported to be hexacoordinated triorganoantimony(V) compounds. Thus, the barrier to displacing both halides in $R_3SbCl_2$ with bidentate ligands appears to be a strong preference for six- rather than seven-coordination by the antimony [14,15].

In their IR spectra, $\nu$(C=O) of the complexes (1604–1612 $cm^{-1}$) have experienced a low wavenumber shift from the position in the respective ligands (1650–1701 $cm^{-1}$). This suggests that the oxygen atoms of the carbonyl groups are involved in coordination to the antimony. [4,6] The broad absorption band due to $\nu$(OH···O) in the ligands has disappeared in the spectra of the **1** and **2**, though such a band remains at 3315 $cm^{-1}$ in **3** due to the $C_6H_4OH$-2 functionality. All three IR spectra show a strong intensity bands between 700–740 $cm^{-1}$ due to the Sb–O–Sb linkage, thereby establishing the anhydride nature of the compounds.[16]

The NMR spectra of the complexes are unexceptional, save to confirm the composition of each species. The singlet due to the enol hydroxyl proton at 9.72–11.38 ppm in the ligands has disappeared in the complexes, as expected. None of the three FAB mass spectra contain the parent ion, but all three show a low abundance fragment which can be assigned $Ph_3SbL^+$. The most intense peak in all cases was due to the ligand, L.

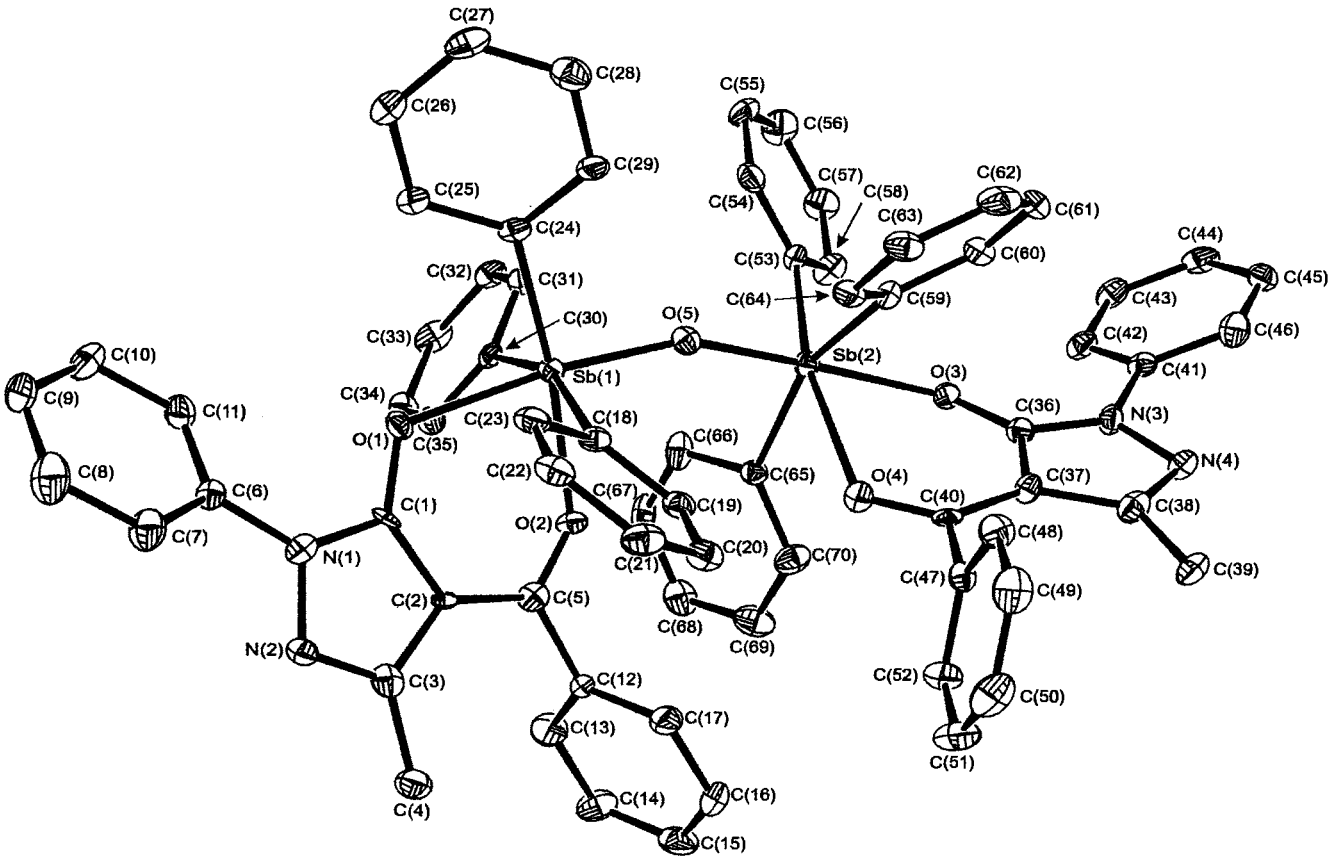



Fig. 1. The asymmetric unit of **2**, showing the labelling scheme used in the text and tables. Thermal ellipsoids are at the 30% probability level.

### *2.2. The structure of μ-oxo-bis[triphenyl(1-phenyl-3-methyl-4-benzoylpyrazolon-5-ato)antimony(V)] (2)*

The asymmetric unit of (**2**) is shown in Fig. 1. The overall coordination about both antimony centres can be described as distorted octahedral $C_3O_3Sb$, with the coordinating atoms present in a mer arrangement around each central atom. Each octahedron shares a common vertex, generating a Sb–O–Sb linkage which, as shown in Fig. 2, is non-linear [$\angle$Sb(1)–O(5)–Sb(2) = 155.6(3)°].

There are three sets of Sb–O bonds of which the bridging Sb(1)–O(5) and Sb(2)–O(5) [1.960(5), 1.946(5) Å] are the shortest, though these are towards the longer end of the range known for $[Ph_3Sb(X)]_2O$ species.[17–20] The average Sb–C bond is also long for such systems [2.135(8) Å] and both these features seem to result from a demand by antimony to achieve octahedral coordination through chelation of the pyrazolone ligand.

The angular Sb–O–Sb linkage in **2** is, perhaps, somewhat surprising given the collective bulk of the phenyl and pyrazolone ligands and the lengthening of Sb–$O_b$ and Sb–C bonds, though it should be noted that this moiety is very flexible in oxo-bridged triorganoantimony(V) species. Known $\angle$Sb–O–Sb range from 135–180° and, remarkably, both extremes of this range are found for $(Ph_3SbI)_2O$ which crystallises in both 'linear' and 'bent' forms [21]. In addition, the angle at the bridging oxygen is by no means a function of steric crowding alone, as reflected in the iodide example above and the marked difference in data for the corresponding chloride (139.0°) and bromide (170.2, 176.6°) complexes [22,23]. In **2**, the two vertex-sharing octahedra are clearly facilitated by a staggering of ligands at either end of the molecule (Fig. 2).

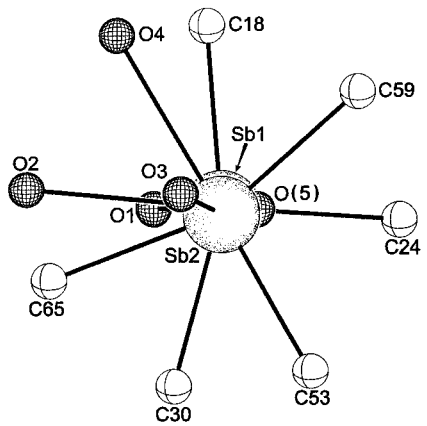
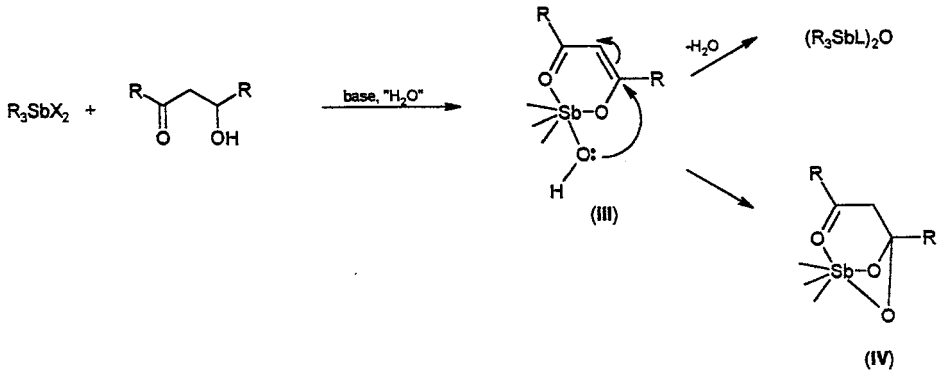



Fig. 2. A view of **2** along the Sb(1)–Sb(2) vector, showing the non-linearity of the Sb(1)–O(5)–Sb(2) bridge and the staggered disposition of the ligands at either end of the molecule.




Scheme 1.

The shorter of the coordinating Sb–O bonds [Sb(1)–O(1) and Sb(2)–O(3)] are associated with the longer C–O bonds [i.e. C(1)–O(1) and C(36)–O(3): 1.314(8) and 1.275(9) Å, respectively] and vice versa for Sb(1)–O(2) and Sb(2)–O(4) [C(5)–O(2) and C(40)–O(4): 1.247(9) and 1.256(9) Å, respectively]. The magnitude of asymmetry of the two chelate C–O bond lengths is more pronounced than that observed in the two Sn–O bonds in the diorganotin(IV) derivatives [4,6] but less than in the phenylmercury(II) derivative [11] of the same ligand. Also, the asymmetry of the two Sb–O bonds is different for the two $C_3O_2Sb$ chelate systems [$D = 0.085$ and 0.184 Å for the pyrazolone chelates of Sb(1) and Sb(2), respectively].

The chelate rings are not planar, but are bent along the O(1)···O(2) and O(3)···O(4) axes by $\varphi =$ 27.6 and 25.7°, respectively. The bite angle of the ligand is 82.0(2) and 78.7(2)° in the $Sb(1)O_2C_3$ and $Sb(2)O_2C_3$ chelates, respectively and are consistent with the average of ca. 80° reported for this ligand in previously reported octahedral 1:2 diorganotin(IV) derivatives [4,6]. The bond distances of the pyrazole rings show evident conjugation comparable with values found in the free ligand [24]. The five atoms of each pyrazole are planar (maximum displacement from least square plane through them is 0.011 Å).

Though the purely inorganic system $[Cl_3Sb(acac)]_2O$ has been crystallographically characterised [25], as far as we are aware the only triorganoantimony $\beta$-diketonate which has been crystallographically authenticated is $(4\text{-}ClC_6H_4)_3Sb[CF_3C(O)_2CH_2C(O)CH_3]$ [26]. This remarkable compound (IV) is formed in reactions of a type described earlier, and which can be speculated as having a common intermediate (III) with the reactions described in this work (Scheme 1). It is noteworthy that only fluorinated $\beta$-diketones produce (IV) rather than $(R_3SbL)_2O$, presumably because they make the ketonic carbon sufficiently electrophilic.

## 3. Experimental

### *3.1. Synthesis of μ-oxo-bis[(1-phenyl-3-methyl-4-acetyl pyrazolon-5-ato)triphenylantimony(V)], [(Ph3SbPMAP)2O] (1)*

Methanolic solutions (total volume 120 ml) of triphenylantimony(V) dichloride (0.42 g, 0.001 mol), potassium hydroxide (0.06 g, 0.001 mol) and HPMAP (0.22 g, 0.001 mol were stirred for 4 h at r.t. The KCl formed was filtered, and the solvent removed in vacuo. The brown product obtained was recrystallised from hot chloroform/toluene (1:1) mixture to yield an oil, which solidified on addition of diethyl ether (30 ml) to give white crystalline product. The product was isolated by filtration and dried under vacuum (0.45 g, 78%), m.p. 196°C. Found (Calc for $C_{60}H_{52}N_4O_5Sb_2$): C, 62.7(62.9); H, 4.6(4.6); N, 4.8(4.9)%. NMR($CDCl_3$, $\delta$ ppm, $J$ Hz): $^1$H, 2.38 (s, 6H, 3-Me), 2.30 (s, 6H, 4-acetyl Me), 7.22–7.75 (m, 40H, $C_6H_5$); $^{13}$C, 16.9(3-Me), 28.5(4-acetyl, Me), 106.4 (C-4), 149.5 (C-3), 161.8 (4-acetyl, CO), 192.6 (C-5), 121.5, 125.3, 127.8, 136.7 (N-$C_6H_5$), 122.6, 127.1, 128.6, 129.1, 129.8, 129.9, 130.3, 134.7, 135.1, ($SbC_6H_5$). Mass spectral fragments (FAB) [relative intensities(%)]: 567(3.0), [$Ph_3Sb(PMAP)^+$]; 215(100.0), [$PMAP^{+\cdot}$]; 352(2.0), [$Ph_3Sb^{+\cdot}$]; 121(1.0), [$Sb^+$].

### *3.2. Synthesis of μ-oxo-bis[(1-phenyl-3-methyl-4-benzoylpyrazolon-5-ato)triphenylantimony(V)], [(Ph3SbPMBP)2O] (2)*

Compound (**2**) was prepared in a manner analogous to that described for (**1**). The yellow product obtained was recrystallised from chloroform/toluene (1:1) mixture, yielding white crystals (60%), m.p. 216°C. Found (Calc. for $C_{70}H_{66}N_4O_5Sb_2$): C, 65.2 (65.3); H, 5.1(5.1); N, 4.5(4.4). NMR($CDCl_3$, $\delta$ ppm, $J$ Hz): $^1$H, 1.75 (s, 6H, 3-Me), 7.24–7.78 (m, 50H, $C_6H_5$); $^{13}$C, 17.2 (3-Me), 127.6, 128.3, 130.9, 129.2

Table 1
Final fractional atomic coordinates ($\times 10^4$) for **2**

| Atom | *x* | *y* | *z* |
|---|---|---|---|
| Sb(1) | 2273(1) | 4979(1) | 1910(1) |
| Sb(2) | 1870(1) | 7386(1) | 2574(1) |
| O(1) | 3166(4) | 3988(4) | 1515(3) |
| O(2) | 4212(4) | 6146(4) | 2076(3) |
| O(3) | 2085(5) | 8894(4) | 3077(3) |
| O(4) | 3190(5) | 7626(4) | 3642(3) |
| O(5) | 1711(4) | 6022(4) | 2262(3) |
| N(1) | 4626(6) | 3397(4) | 1784(3) |
| N(2) | 5798(6) | 3744(4) | 2115(4) |
| N(3) | 1995(6) | 9945(5) | 4093(3) |
| N(4) | 2281(6) | 10131(4) | 4865(3) |
| C(1) | 4235(6) | 4148(5) | 1806(4) |
| C(2) | 5167(6) | 5027(5) | 2145(4) |
| C(3) | 6116(7) | 4697(6) | 2337(4) |
| C(4) | 7290(7) | 5276(6) | 2726(5) |
| C(5) | 5110(7) | 5983(5) | 2252(4) |
| C(7) | 4391(5) | 1660(4) | 1707(3) |
| C(8) | 3925(6) | 686(3) | 1346(3) |
| C(9) | 3158(5) | 448(3) | 711(3) |
| C(10) | 2857(5) | 1183(4) | 438(3) |
| C(11) | 3323(5) | 2157(3) | 799(3) |
| C(6) | 4090(5) | 2395(3) | 1434(3) |
| C(13) | 7073(5) | 7407(4) | 2111(2) |
| C(14) | 8040(4) | 8280(4) | 2396(3) |
| C(15) | 8135(4) | 8636(3) | 3143(3) |
| C(16) | 7263(5) | 8119(4) | 3605(2) |
| C(17) | 6296(4) | 7246(4) | 3320(2) |
| C(12) | 6201(4) | 6890(3) | 2572(3) |
| C(19) | 3298(4) | 5445(2) | 3564(3) |
| C(20) | 3612(5) | 5213(3) | 4242(2) |
| C(21) | 3344(5) | 4218(4) | 4350(2) |
| C(22) | 2762(5) | 3454(3) | 3780(3) |
| C(23) | 2448(4) | 3685(3) | 3101(2) |
| C(18) | 2716(4) | 4680(3) | 2993(2) |
| C(25) | 371(4) | 2879(4) | 1266(3) |
| C(26) | −747(5) | 2081(3) | 1171(3) |
| C(27) | −1698(4) | 2143(3) | 1550(3) |
| C(28) | −1532(4) | 3001(4) | 2024(3) |
| C(29) | −414(4) | 3799(3) | 2119(3) |
| C(24) | 537(3) | 3738(3) | 1740(3) |
| C(31) | 1588(3) | 5594(4) | 478(2) |
| C(32) | 1714(4) | 5938(4) | −216(2) |
| C(33) | 2752(5) | 6080(4) | −579(2) |
| C(34) | 3662(4) | 5879(4) | −247(2) |
| C(35) | 3536(4) | 5535(4) | 447(2) |
| C(30) | 2498(4) | 5392(3) | 810(2) |
| C(36) | 2274(7) | 9201(5) | 3768(4) |
| C(37) | 2793(7) | 8859(5) | 4358(4) |
| C(38) | 2764(8) | 9494(6) | 5012(4) |
| C(39) | 3224(8) | 9542(6) | 5791(4) |
| C(40) | 3196(6) | 8085(5) | 4265(4) |
| C(42) | 1367(4) | 10 570(4) | 3026(2) |
| C(43) | 710(5) | 11 053(4) | 2724(2) |
| C(44) | 45(5) | 11 432(4) | 3175(3) |
| C(45) | 37(4) | 11 328(4) | 3929(3) |
| C(46) | 694(5) | 10 845(4) | 4231(2) |
| C(41) | 1359(4) | 10 465(3) | 3779(3) |
| C(48) | 2856(3) | 7137(4) | 5376(3) |
| C(49) | 3288(5) | 6828(4) | 5981(3) |
| C(50) | 4516(5) | 7132(4) | 6125(3) |
| C(51) | 5311(4) | 7745(4) | 5664(3) |
| C(52) | 4879(4) | 8053(4) | 5060(3) |
| C(47) | 3651(4) | 7750(4) | 4916(2) |
| C(54) | −289(4) | 6649(3) | 1440(3) |
| C(55) | −1061(4) | 6715(3) | 889(3) |
| C(56) | −849(5) | 7635(4) | 632(3) |
| C(57) | 137(5) | 8489(3) | 925(3) |
| C(58) | 910(4) | 8423(3) | 1476(3) |
| C(53) | 697(4) | 7503(3) | 1733(2) |
| C(60) | −53(5) | 7291(3) | 3686(3) |
| C(61) | −836(4) | 6892(4) | 4227(3) |
| C(62) | −915(4) | 6005(4) | 4491(3) |
| C(63) | −212(5) | 5516(3) | 4212(3) |
| C(64) | 571(4) | 5914(3) | 3671(3) |
| C(59) | 650(4) | 6802(3) | 3407(2) |
| C(66) | 3556(4) | 7948(3) | 1351(2) |
| C(67) | 4609(5) | 8412(4) | 1005(2) |
| C(68) | 5640(4) | 9074(4) | 1422(3) |
| C(69) | 5617(4) | 9273(4) | 2185(3) |
| C(70) | 4564(4) | 8810(4) | 2531(2) |
| C(65) | 3533(4) | 8147(4) | 2114(2) |

(4-$C_6H_5CO$) 105.7 (C-4), 149.6 (C-3), 161.5 (4-benzoyl, CO), 191.3 (C-5), 121.6, 125.0, 128.1, 138.0 (N-$C_6H_5$) 121.6, 125.0, 128.1, 138.0 ($C_6H_5Sb$). Mass spectral fragments (FAB) [relative intensities(%)]: 629(2.0), [$Ph_3Sb(PMBP)^+$]; 279(100.0), [$PMBP^+$]; 121(3.0), [$Sb^{+\cdot}$].

### *3.3. Synthesis of μ-oxo-bis[1-salicyloyl-3-methyl-pyrazolon-5-ato]triphenylantimony(V), [(Ph$_3$SbSPz)$_2$O] (3)*

Triphenylantimony(V) dichloride (0.42 g, 0.001 mol) in dry THF (20 ml) was added to a suspension of SPzH (0.22 g, 0.001 mol) and sodium methoxide (0.05 g, 0.001 mol) in methanol (20 ml). The mixture was stirred for 4 h at r.t. The solution was then allowed to evaporate slowly at r.t. for 2 days, during which a crude yellow product was obtained and which was subsequently recrystallised from warm acetonitrile (0.18 g, 40%), m.p. 220°C. Found (Calc. for $C_{58}H_{48}N_4O_7Sb$): C, 60.9(61.0); H, 4.3(4.2); N, 5.0(4.9)%. NMR(DMSO-$d_6$, $\delta$ ppm, *J* Hz): $^1$H, 2.48 (s, 6H, 3-Me), 5.83 (s, 2H, C-4 methine H), 7.34–7.87 (m, 38H, $C_6H_5$), 11.72 (s, 2H, salicyloyl OH); $^{13}$C, 18.8 (3-Me), 142.1 (C-3), 107.0 (C-4), 152.7 (C-5), 164.4 (salicyloyl, CO), 87.2, 117.2, 128.7, 129.4, 129.5, 131.9, 136.2 (salicyloyl phenyl), 128.3, 128.4, 129.3, 129.5, 130.0, 130.1, 132.2, 132.6, 133.3, 138.6 ($C_6H_5Sb$). Mass spectral fragments (FAB) [relative intensities(%)]: 569(4.0), [$Ph_3Sb(SPz)^+$]; 492(4.0), [$Ph_2Sb(SPz)^{+\cdot}$]; 415(14.0), [$PhSb(SPz)^+$]; 337(22.0), [$Sb(SPz)^{+\cdot}$]; 217(100.0), [$SPz^{+\cdot}$]; 352(18.0), [$Ph_3Sb^{+\cdot}$]; 274(26.0), [$Ph_2Sb^+$]; 121(10.0), [$Sb^+$].

Table 2
Selected geometric data (Å, °) for **2**

| | | | |
|---|---|---|---|
| Bond distances (Å) | | | |
| Sb(1)–O(5) | 1.960(5) | Sb(2)–C(53) | 2.127(4) |
| Sb(1)–C(30) | 2.135(3) | Sb(2)–C(59) | 2.140(3) |
| Sb(1)–C(18) | 2.153(3) | Sb(2)–O(3) | 2.208(5) |
| Sb(1)–C(24) | 2.161(4) | Sb(2)–O(4) | 2.392(5) |
| Sb(1)–O(1) | 2.205(5) | O(1)–C(1) | 1.314(8) |
| Sb(1)–O(2) | 2.290(5) | O(2)–C(5) | 1.247(9) |
| Sb(2)–O(5) | 1.946(5) | O(3)–C(36) | 1.275(9) |
| Sb(2)–C(65) | 2.125(4) | O(4)–C(40) | 1.256(9) |
| Bond Angles (°) | | | |
| O(5)–Sb(1)–C(30) | 94.6(2) | O(5)–Sb(2)–C(65) | 97.3(2) |
| O(5)–Sb(1)–C(18) | 96.1(2) | O(5)–Sb(2)–C(53) | 98.6(2) |
| C(30)–Sb(1)–C(18) | 159.5(2) | C(65)–Sb(2)–C(53) | 99.1(2) |
| O(5)–Sb(1)–C(24) | 97.3(2) | O(5)–Sb(2)–C(59) | 88.3(2) |
| C(30)–Sb(1)–C(24) | 99.8(2) | C(65)–Sb(2)–C(59) | 158.1(2) |
| C(18)–Sb(1)–C(24) | 96.1(2) | O(5)–Sb(2)–O(3) | 172.5(2) |
| O(5)–Sb(1)–O(1) | 171.6(2) | C(65)–Sb(2)–O(3) | 85.5(2) |
| C(30)–Sb(1)–O(1) | 82.8(2) | C(53)–Sb(2)–O(3) | 87.8(2) |
| C(18)–Sb(1)–O(1) | 84.1(2) | C(59)–Sb(2)–O(3) | 86.6(2) |
| C(24)–Sb(1)–O(1) | 91.1(2) | O(5)–Sb(2)–O(4) | 94.9(2) |
| O(5)–Sb(1)–O(2) | 89.7(2) | C(65)–Sb(2)–O(4) | 81.0(2) |
| C(30)–Sb(1)–O(2) | 81.1(2) | C(53)–Sb(2)–O(4) | 166.4(2) |
| C(18)–Sb(1)–O(2) | 81.4(2) | C(59)–Sb(2)–O(4) | 77.5(2) |
| C(24)–Sb(1)–O(2) | 172.9(2) | O(3)–Sb(2)–O(4) | 78.7(2) |
| O(1)–Sb(1)–O(2) | 82.0(2) | | |

### 3.4. X-Ray data collection and structure determination of (**2**)

Crystals of (**2**) were grown by slow crystallisation from toluene/chloroform (1:2); one of approximate dimensions $0.4 \times 0.4 \times 0.15$ mm was mounted in a glass capillary and used for data collection.

Crystal data; $C_{70}H_{56}N_4O_5Sb_2$, $M = 1276.7$; triclinic; space group $P\bar{1}$; $a = 12.169(6)$; $b = 14.569(4)$, $c = 18.149(6)$Å; $\alpha = 95.47(3)$, $\beta = 91.49(3)$, $\gamma = 113.25(4)°$, $U = 2935.7$ Å$^3$, $Z = 2$, $D_{calc} = 1.44$ g cm$^{-3}$, $\mu$(Mo–K$_\alpha$) = 9.76 cm$^{-1}$, $F(000) = 1292$. Data were measured at 170°K on a CAD4 automatic four-circle diffractometer in the range $2 \leq \theta \leq 24°$. Reflections numbering 9670 were collected, of which 5581 were unique with $I \geq 2\sigma(I)$. Data were corrected for Lorentz and polarization, and also for absorption (maximum, minimum absorptions corrections; 1.145 and 0.748, respectively). The structure was solved by Patterson methods and refined using the SHELX suite programs. [27,28] In the final least squares cycles all atoms were allowed to vibrate anisotropically. Phenyl rings were treated as rigid hexagons and hydrogen atoms were included at calculated positions where appropriate. Final residuals after ten cycles of least squares [i.e. based on 5562 $F^2$ data with $Fo > 4\sigma(Fo)$] were $R_1 = 0.0491$, $wR_2 = 0.1272$. Goodness-of-fit = 0.969. Maximum final shift/estimated S.D. = 0.002. The maximum and minimum residual densities were 0.82 and $-0.85$ e Å$^{-3}$, respectively.

Final fractional atomic coordinates, selected bond distances and angles are given in Tables 1 and 2, respectively. Tables of anisotropic temperature factors are available as supplementary data. The asymmetric unit along with the labelling scheme used and the view along the SbOSb linkage are shown in Figs. 1 and 2, respectively. Figures were produced using ORTEX. [29]

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## References

[1] B.S. Jensen, Acta Chem. Scand. 13 (1959) 1347.
[2] F. Bonati, L.A. Oro, M.T. Phillips, Polyhedron 4 (1985) 357.
[3] S. Saxena, R. Bohra, A.K. Rai, Inorg. Chim. Acta 173 (1990) 191.
[4] C. Pettinari, G. Rafaini, C.G. Lobbia, A. Lorenzotti, B. Bovio, J. Organomet. Chem. 405 (1991) 75.
[5] C. Pettinari, F. Marchetti, A. Cingolani, D. Leonesi, G.G. Lobbia, A. Lorenzotti, J. Organomet. Chem. 454 (1993) 59.
[6] B. Bovio, A. Cingolani, F. Marchetti, C. Pettinari, J. Organomet. Chem. 458 (1993) 39.
[7] C. Pettinari, F. Marchetti, D. Leonesi, M. Rossi, F. Caruso, J. Organomet. Chem. 483 (1994) 123.
[8] C. Pettinari, F. Marchetti, A. Cingolani, C. Marciante, R. Spagna, M. Colapietro, Polyhedron 13 (1994) 939.
[9] F. Marchetti, C. Pettinari, A. Cingolani, G.G. Lobbia, A. Cassetta, L. Barba, J. Organomet. Chem. 517 (1996) 141.
[10] M.F. Mahon, K.C. Molloy, B.A. Omotowa, M.A. Mesubi, J. Organomet. Chem. 511 (1996) 227.
[11] M.F. Mahon, K.C. Molloy, B.A. Omotowa, M.A. Mesubi, J. Organomet. Chem. 525 (1996) 239.
[12] A. Ouchi, H. Honda, S. Kitazima, J. Inorg. Nucl. Chem. 37 (1975) 2559.
[13] S. Kraft, M. Weiber, Z. Anorg. Allg. Chem. 607 (1992) 153.
[14] K. Singhal, A.K. Aggarwal, P. Raj, Ind. J. Chem. 31A (1992) 797.
[15] J.W. Siebert, S.J. Lange, C. Stern, B.M. Hoffmann, A.G.M. Barrett, J. Chem. Soc. Chem. Commun. (1994) 1751.
[16] C. Silvestru, I. Haiduc, J. Organomet. Chem. 365 (1989) 83.
[17] G. Ferguson, D.R. Riley, Acta Crystallogr. B29 (1973) 2221.
[18] Z.A. Satrikova, T.M. Shchegoleva, V.K. Trunov, J.E. Pokrovskaya, Sov. Phys. Crystallogr. 23 (1978) 547.
[19] H. Preut, R. Ruther, F. Huber, Acta Crystallogr. C41 (1985) 358.
[20] H. Preut, R. Ruther, F. Huber, Acta Crystallogr. C42 (1986) 1154.
[21] M.J. Taylor, L.-J. Baker, C.E.F. Rickard, P.W.J. Surman, J. Organomet. Chem. 498 (1995) 14.
[22] E.R.T. Tiekink, J. Organomet. Chem. 333 (1987) 199.
[23] A. Ouchi, S. Sato, Bull. Chem. Soc. Jpn. 61 (1988) 1806.
[24] M.J. O'Connell, C.G. Ramsey, P. Steel, Aust. J. Chem. 38 (1985) 401.

[25] S. Blosl, W. Schwarz, A. Schmidt, Z. Naturforsch. 34b (1979) 1711.
[26] F. Ebina, T. Uehiro, T. Iwamoto, A. Ouchi, Y. Yoshino, J. Chem. Soc. Chem. Commun. (1976) 245.
[27] G.M. Sheldrick, SHELX86, A Program for crystal structure determination, University of Göttingen, Göttingen, 1986.
[28] G.M. Sheldrick, SHELX93, A Program for crystal structure determination, University of Göttingen, Göttingen, 1993.
[29] P. McArdle, J. Appl. Cryst. 27 (1994) 438.