Tris(p-chlorophenyl)-5,5,5-trifluoro-4,4-dihydroxy-2-pentanonatoantimony(V)

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Abstract. $SbC_{23}H_{17}O_3F_3Cl$, monoclinic, $P2_1/c$, $a = 11.518$ (1), $b = 20.962$ (2), $c = 12.259$ (1) Å, $\beta = 124.08$ (1)°, $Z = 4$, $D_m = 1.68$, $D_x = 1.70$ g cm^{-3}. The fluorinated β-diketone is hydrated to form a novel terdentate ligand: a 4,4-*gem*-dihydroxy,2-one dianion. The Sb atom has a distorted octahedral coordination, being surrounded by three O atoms of the new ligand and three C atoms of the aryl groups in facial positions.

Introduction. Fluorinated β-diketone, acting as a bidentate ligand in organoantimony(V) compounds, was found to change into a novel type of ligand in moist organic solvent, and its fundamental structure was reported (Ebina, Uehiro, Iwamoto, Ouchi & Yoshino, 1976). The detailed structure will be described here.

Colourless plate-like crystals were obtained by recrystallization from a mixture of chloroform and petroleum spirit. Intensities and cell dimensions were measured on a Rigaku automated four-circle diffractometer with a crystal of dimensions $0.4 \times 0.4 \times 0.05$ mm. Data were collected by the θ–2θ scan technique with graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0.7107$ Å). Of the 4518 independent reflexions measured in the range $4 < 2\theta < 50°$, 2655 had reliable

intensities with $|F| > 3\sigma(|F|)$. The intensities were corrected for Lorentz–polarization factors. No corrections were made for absorption ($\mu = 14.27$ cm^{-1}).

Table 2. *Interatomic distances* (Å) *with estimated standard deviations in parentheses*

Sb—O(1)	2.534 (8)	C(14)—C(15)	1.428 (21)
Sb—O(2)	2.036 (7)	C(15)—C(16)	1.423 (14)
Sb—O(3)	2.032 (9)	C(16)—C(11)	1.338 (19)
Sb—C(11)	2.142 (8)	C(14)—Cl(1)	1.742 (10)
Sb—C(21)	2.113 (10)	C(21)—C(22)	1.346 (19)
Sb—C(13)	2.107 (15)	C(22)—C(23)	1.404 (17)
O(1)—C(2)	1.213 (17)	C(23)—C(24)	1.414 (22)
O(2)—C(4)	1.403 (17)	C(24)—C(25)	1.347 (20)
O(3)—C(4)	1.432 (13)	C(25)—C(26)	1.360 (16)
C(1)—C(2)	1.511 (20)	C(26)—C(21)	1.429 (20)
C(2)—C(3)	1.510 (20)	C(24)—Cl(2)	1.747 (12)
C(3)—C(4)	1.530 (16)	C(31)—C(32)	1.384 (16)
C(4)—C(5)	1.535 (17)	C(32)—C(33)	1.406 (23)
C(5)—F(1)	1.337 (20)	C(33)—C(34)	1.388 (16)
C(5)—F(2)	1.317 (14)	C(34)—C(35)	1.408 (18)
C(5)—F(3)	1.353 (14)	C(35)—C(36)	1.394 (22)
C(11)—C(12)	1.430 (18)	C(36)—C(31)	1.422 (13)
C(12)—C(13)	1.426 (13)	C(34)—Cl(3)	1.725 (18)
C(13)—C(14)	1.326 (20)		

Table 1. *Final atomic coordinates* ($\times 10^4$) *with estimated standard deviations in parentheses*

	x	y	z		x	y	z
Sb	3321 (1)	1559 (0)	3398 (1)	C(16)	673 (12)	2291 (7)	1480 (11)
O(1)	2180 (8)	491 (4)	2416 (7)	Cl(1)	−2038 (4)	2550 (2)	−2381 (3)
O(2)	4783 (7)	887 (4)	4546 (6)	C(21)	4434 (10)	2424 (5)	4159 (9)
O(3)	4365 (7)	1217 (4)	2626 (6)	C(22)	4245 (11)	2833 (6)	4894 (10)
C(1)	2125 (17)	−611 (7)	1975 (16)	C(23)	4936 (12)	3421 (6)	5360 (10)
C(2)	2842 (13)	29 (6)	2462 (11)	C(24)	5928 (11)	3556 (6)	5056 (10)
C(3)	4384 (12)	56 (6)	3002 (11)	C(25)	6143 (12)	3159 (6)	4322 (10)
C(4)	5009 (11)	713 (6)	3574 (10)	C(26)	5436 (11)	2597 (6)	3872 (10)
C(5)	6587 (11)	729 (6)	4161 (11)	Cl(2)	6837 (5)	4278 (2)	5620 (4)
F(1)	6853 (8)	584 (5)	3259 (8)	C(31)	2400 (11)	1404 (5)	4452 (9)
F(2)	7169 (7)	1283 (4)	4697 (8)	C(32)	1041 (11)	1184 (7)	3858 (10)
F(3)	7254 (7)	274 (4)	5097 (7)	C(33)	503 (12)	980 (7)	4584 (11)
C(11)	1570 (10)	1870 (5)	1521 (9)	C(34)	1407 (12)	1003 (7)	5946 (12)
C(12)	1430 (11)	1610 (6)	375 (10)	C(35)	2767 (12)	1259 (7)	6587 (11)
C(13)	270 (11)	1856 (6)	−837 (10)	C(36)	3313 (10)	1405 (7)	5849 (9)
C(14)	−617 (12)	2275 (6)	−875 (11)	Cl(3)	783 (4)	772 (2)	6878 (4)
C(15)	−476 (13)	2539 (7)	267 (11)				

Table 3. *Bond angles (°) with estimated standard deviations in parentheses*

O(1)—Sb—O(2)	74·0 (3)	C(16)—C(11)—C(12)	123·7 (10)
O(1)—Sb—O(3)	75·6 (3)	C(11)—C(12)—C(13)	114·2 (11)
O(1)—Sb—C(11)	80·4 (3)	C(12)—C(13)—C(14)	122·0 (12)
O(1)—Sb—C(21)	173·5 (3)	C(13)—C(14)—C(15)	124·0 (11)
O(1)—Sb—C(31)	81·3 (4)	C(13)—C(14)—Cl(1)	120·3 (10)
O(2)—Sb—O(3)	66·5 (3)	C(14)—C(15)—C(16)	114·3 (12)
O(2)—Sb—C(21)	103·1 (3)	C(15)—C(16)—C(11)	121·7 (12)
O(2)—Sb—C(31)	88·6 (4)	Sb—C(21)—C(22)	118·9 (9)
O(3)—Sb—C(11)	93·1 (3)	C(26)—C(21)—C(22)	117·3 (11)
O(3)—Sb—C(21)	97·9 (4)	C(21)—C(22)—C(23)	124·3 (13)
C(11)—Sb—C(31)	102·2 (5)	C(22)—C(23)—C(24)	115·2 (13)
Sb—O(1)—C(2)	122·5 (8)	C(23)—C(24)—C(25)	122·1 (12)
Sb—O(2)—C(4)	93·9 (7)	C(23)—C(24)—Cl(2)	117·5 (11)
Sb—O(3)—C(4)	93·2 (6)	C(24)—C(25)—C(26)	120·8 (14)
O(1)—C(2)—C(1)	120·0 (13)	C(25)—C(26)—C(21)	120·2 (13)
O(1)—C(2)—C(3)	123·1 (12)	Sb—C(31)—C(36)	116·9 (8)
C(1)—C(2)—C(3)	117·0 (12)	C(36)—C(31)—C(32)	119·3 (12)
C(2)—C(3)—C(4)	111·7 (11)	C(31)—C(32)—C(33)	122·6 (11)
O(2)—C(4)—O(3)	103·8 (9)	C(32)—C(33)—C(34)	116·9 (12)
O(2)—C(4)—C(5)	109·3 (8)	C(33)—C(34)—C(35)	122·3 (14)
O(3)—C(4)—C(3)	113·3 (8)	C(33)—C(34)—Cl(3)	118·5 (10)
O(3)—C(4)—C(5)	107·6 (10)	C(34)—C(35)—C(36)	119·2 (11)
C(3)—C(4)—C(5)	111·4 (10)	C(35)—C(36)—C(31)	118·9 (11)
Sb—C(11)—C(12)	117·4 (8)		

Fig. 1. A perspective drawing of the title compound.

The structure was solved by the heavy-atom method, and refined by a block-diagonal least-squares program. The final refinement was carried out with the full-matrix least-squares program *LINUS* (Coppens & Hamilton, 1970), with anisotropic temperature factors for all non-hydrogen atoms. The atomic scattering factors and the corrections for anomalous scattering were taken from *International Tables for X-ray Crystallography* (1974). Unit weight was given to all reflexions. Final R value was 0·059. H atoms were not included in the calculations.

The final atomic coordinates, the interatomic distances and the bond angles with their estimated standard deviations are listed in Tables 1,* 2 and 3 respectively.

Discussion. A perspective drawing of the complex including the numbering scheme is shown in Fig. 1. The fluorinated β-diketone precursor acting as a bidentate ligand is hydrated at the C(3) and C(4) atoms, which are transformed into methylene and *gem*-dihydroxy C. C(4) is bonded tetrahedrally to two O and two C atoms. The mean bond length between C(4) and the alcohol O atoms is 1·418 (15) Å. The distances on both sides of C(3), C(3)—C(2) and C(3)—C(4), are normal for single bonds, indicating that C(3) is a methylene C atom. The C(2)—O(1) distance is typical of a carbonyl group.

The coordination around Sb is a distorted octahedron. The new ligand behaves as a terdentate,

bonding to Sb through two hydroxy O atoms and a carbonyl O. They occupy facial positions in the coordination. If the orientation of the three aryl rings is ignored, the molecule has a pseudo symmetry plane comprised of the atoms of the dihydroxy ketone skeleton, except O(2) and O(3), Sb and C(21). The plane is approximately perpendicular to the plane formed by O(2), O(3), C(11) and C(31), the dihedral angle being about 88°. The distances Sb—C(11), —C(21) and —C(31), Sb—O(2) and —O(3) are equivalent within the experimental errors. While Sb—O(1) is longer than the other metal—donor atom distances, a weak interaction may exist because $\nu_{C=O}$ is shifted to a lower wavenumber region in the IR spectrum. The O(2)—Sb—O(3) bond angle is similar to that of carbonato and dithiocarbamato complexes (Ferguson & Hawley, 1974; Raston & White, 1976).

No remarkable interaction between the molecules is observed. The shortest intermolecular distance between non-hydrogen atoms is 3·121 (19) Å [C(2)···F(3)].

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32786 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.
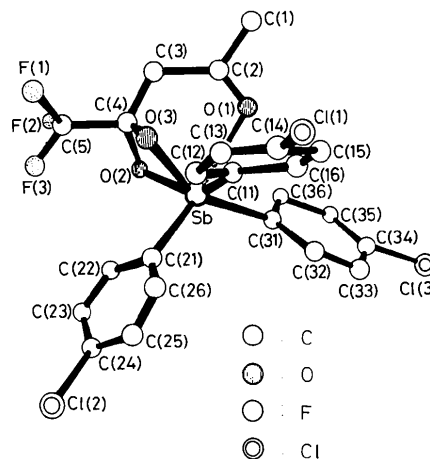
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