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# Structures of $\mu$-Oxo-bis[(benzenesulfonato)triphenylantimony(V)] and $\mu$-Oxo-bis[(trifluoromethylsulfonato)triphenylantimony(V)]

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**Abstract.** (I) $[(C_6H_5)_3SbO_3SC_6H_5]_2O$, $M_r = 1036{\cdot}5$, monoclinic, $P2_1/c$, $a = 16{\cdot}142\,(6)$, $b = 17{\cdot}733\,(6)$, $c = 16{\cdot}141\,(6)$ Å, $\beta = 108{\cdot}28\,(5)°$, $V = 4387$ Å$^3$, $Z = 4$, $D_x = 1{\cdot}569$ Mg m$^{-3}$, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71069$ Å, $\mu = 1{\cdot}39$ mm$^{-1}$, $F(000) = 2072$, $T = 292\,(1)$ K, $R = 0{\cdot}044$ for 3712 observed reflexions $[I > 3\sigma(I)]$. (II) $[(C_6H_5)_3SbO_3SCF_3]_2O$, $M_r = 1020{\cdot}3$, monoclinic, $P2_1/c$, $a = 20{\cdot}596\,(8)$, $b = 10{\cdot}296\,(5)$, $c = 19{\cdot}546\,(9)$ Å, $\beta = 108{\cdot}24\,(5)°$, $V = 3937$ Å$^3$, $Z = 4$, $D_x = 1{\cdot}721$ Mg m$^{-3}$, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71069$ Å, $\mu = 1{\cdot}57$ mm$^{-1}$, $F(000) = 2008$, $T = 292\,(1)$ K, $R = 0{\cdot}055$ for 2652 observed reflexions $[I > 3\sigma(I)]$. Two ($\mu$-O)—SbPh$_3$—O—S(O$_2$)$R$ [$R = C_6H_5$ (I), $CF_3$ (II)] units with slightly distorted trigonal-bipyramidal surroundings at the Sb atoms – O atoms in apical and C(phenyl) atoms in equatorial positions – are linked *via* a bent Sb—O—Sb bridge in the molecules (I) [139·8 (3)°] and (II) [136·5 (5)°]. The ($\mu$-O)—Sb bond lengths are in the range 1·937 (10) to 1·980 (8) Å. The Sb—O bonds to the unidentately coordinated sulfonate groups are in the range 2·247 (5) to 2·37 (1) Å and appear to have appreciably high ionic character.

**Introduction.** Only rather few structures of distiboxans are known, and the question why the Sb—O—Sb bridge in such compounds is bent in some examples [$(Ph_3SbN_3)_2O$ (Ferguson & Ridley, 1973); $(Me_3SbX)_2O$, $X = ClO_4$, Cl and $N_3$ (Ferguson, March & Ridley, 1975)] or linear in others [$(Ph_3SbOO$—*tert*-$C_4H_9)_2O$ (Starikova, Shchegoleva, Trunov & Pokrovskaya, 1978)] still waits to be answered. We recently reported as a first structure of $(R_3SbO_3SR')_2O$ compounds ($R$, $R'$ = organic groups) that of $(Ph_3SbO_3SCH_2CH_2OH)_2O$, in which we found a linear bridge (Preut, Rüther & Huber, 1985). We now describe the structures of two other compounds of the same formula type, which however have a bent Sb—O—Sb bridge.

**Experimental.** (I) and (II) were synthesized by stirring 5 mmol $Ph_3SbO$ and 5 mmol of the appropriate sulfonic acid in 20 ml acetone at room temperature for 1 h. The precipitates were recrystallized from chloroform by addition of petroleum ether. Approximate dimensions (I) 0·29 × 0·30 × 0·32 mm, (II) 0·37 × 0·06 × 0·10 mm; CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\omega/2\theta$ scan technique, scan speed (I) 0·90–2·5° $\min^{-1}$ and (II) 2·5° $\min^{-1}$; cell parameters from 25 reflexions up to (I) $2\theta = 27{\cdot}8°$ and (II) $2\theta = 26{\cdot}2°$ and equally distributed in reciprocal space; 5100 (I) and 2907 (II) unique reflexions measured in the ranges (I) $1{\cdot}5 \le \theta \le 20{\cdot}8°$, $-16 \le h \le 16$, $0 \le k \le 17$, $0 \le l \le 16$ and (II) $1 \le \theta \le 23°$, $-21 \le h \le 21$, $0 \le k \le 11$, $0 \le l \le 22$; three standard reflexions recorded every 2·5 h remained stable; intensities corrected for Lorentz–polarization effects and absorption *via* $\psi$ scans; max./min. transmission: (I) 1·00/0·94, (II) 1·00/0·94; systematic absences $h0l$, $l = 2n+1$, $0k0$, $k = 2n+1$, space group

Table 1. *Fractional atomic coordinates and* $B_{eq}$, $B$ ($Å^2 \times 8\pi^2$) *for* $(Ph_3SbO_3SC_6H_5)_2O$ (I) *and* $(Ph_3SbO_3SCF_3)_2O$ (II)

$$B_{eq} = \tfrac{4}{3}\textstyle\sum_i \sum_j \beta_{ij} \mathbf{a}_i . \mathbf{a}_j .$$

$(Ph_3SbO_3SC_6H_5)_2O$ (I)

| | $x$ | $y$ | $z$ | $B_{eq}$, $B$ |
|---|---|---|---|---|
| Sb(1) | 0·12297 (4) | −0·11652 (4) | 0·23556 (4) | 2·88 (1) |
| Sb(2) | 0·29865 (4) | −0·14440 (4) | 0·13772 (4) | 3·28 (2) |
| S(1) | 0·0340 (2) | −0·1285 (2) | 0·3965 (2) | 3·24 (6) |
| S(2) | 0·5210 (2) | −0·1294 (2) | 0·1412 (2) | 5·22 (8) |
| O(1) | 0·1819 (4) | −0·1463 (4) | 0·1512 (4) | 3·5 (1)* |
| O(11) | 0·0497 (4) | −0·0802 (4) | 0·3264 (4) | 3·4 (1)* |
| O(12) | 0·0780 (5) | −0·0979 (4) | 0·4806 (5) | 4·7 (2)* |
| O(13) | 0·0498 (5) | −0·2064 (4) | 0·3827 (5) | 4·7 (2)* |
| O(21) | 0·4279 (4) | −0·1531 (4) | 0·1090 (4) | 4·4 (2)* |
| O(22) | 0·5643 (6) | −0·1661 (6) | 0·2230 (6) | 7·9 (2)* |
| O(23) | 0·5310 (6) | −0·0495 (6) | 0·1384 (6) | 8·4 (3)* |
| C(11) | 0·2278 (6) | −0·1458 (6) | 0·3467 (6) | 3·2 (2)* |
| C(12) | 0·2593 (7) | −0·0911 (7) | 0·4109 (7) | 4·5 (2)* |
| C(13) | 0·3353 (7) | −0·1094 (7) | 0·4824 (7) | 5·1 (3)* |
| C(14) | 0·3714 (8) | −0·1802 (7) | 0·4866 (8) | 5·4 (3)* |
| C(15) | 0·3392 (8) | −0·2340 (7) | 0·4240 (8) | 5·3 (3)* |
| C(16) | 0·2638 (7) | −0·2167 (6) | 0·3502 (7) | 4·1 (2)* |
| C(21) | 0·0156 (6) | −0·1852 (6) | 0·1706 (6) | 3·6 (2)* |
| C(22) | 0·0303 (7) | −0·2581 (7) | 0·1497 (7) | 4·8 (3)* |
| C(23) | −0·0431 (9) | −0·3007 (8) | 0·0977 (9) | 6·4 (3)* |
| C(24) | −0·1241 (8) | −0·2673 (8) | 0·0700 (8) | 6·1 (3)* |
| C(25) | −0·1376 (8) | −0·1969 (8) | 0·0920 (8) | 5·7 (3)* |
| C(26) | −0·0664 (7) | −0·1518 (7) | 0·1451 (7) | 4·5 (2)* |
| C(31) | 0·1059 (6) | −0·0041 (6) | 0·1940 (6) | 3·2 (2)* |
| C(32) | 0·1087 (7) | 0·0553 (7) | 0·2508 (7) | 4·6 (2)* |
| C(33) | 0·1020 (8) | 0·1292 (8) | 0·2168 (8) | 6·1 (3)* |
| C(34) | 0·0893 (9) | 0·1411 (8) | 0·1267 (9) | 6·9 (3)* |
| C(35) | 0·0860 (8) | 0·0805 (7) | 0·0731 (8) | 5·6 (3)* |
| C(36) | 0·0948 (7) | 0·0072 (7) | 0·1057 (7) | 4·5 (2)* |
| C(41) | −0·0792 (6) | −0·1183 (6) | 0·3810 (6) | 3·1 (2)* |
| C(42) | −0·1372 (7) | −0·1633 (6) | 0·3187 (7) | 4·3 (2)* |
| C(43) | −0·2270 (7) | −0·1563 (7) | 0·3057 (8) | 5·2 (3)* |
| C(44) | −0·2554 (7) | −0·1051 (7) | 0·3549 (7) | 5·0 (3)* |
| C(45) | −0·1974 (7) | −0·0604 (7) | 0·4181 (7) | 4·9 (3)* |
| C(46) | −0·1066 (6) | −0·0659 (6) | 0·4295 (6) | 3·8 (2)* |
| C(51) | 0·2518 (6) | −0·1014 (6) | 0·0099 (6) | 3·7 (2)* |
| C(52) | 0·3001 (8) | −0·0476 (7) | −0·0173 (8) | 5·2 (3)* |
| C(53) | 0·2658 (9) | −0·0173 (8) | −0·1010 (9) | 6·9 (3)* |
| C(54) | 0·1846 (9) | −0·0415 (8) | −0·1564 (9) | 6·9 (3)* |
| C(55) | 0·1386 (8) | −0·0964 (8) | −0·1283 (8) | 6·3 (3)* |
| C(56) | 0·1712 (7) | −0·1276 (7) | −0·0437 (7) | 4·5 (2)* |
| C(61) | 0·3596 (6) | −0·0623 (6) | 0·2326 (6) | 3·7 (2)* |
| C(62) | 0·3279 (7) | 0·0090 (7) | 0·2182 (7) | 4·9 (3)* |
| C(63) | 0·3675 (9) | 0·0647 (8) | 0·2837 (8) | 6·3 (3)* |
| C(64) | 0·4351 (8) | 0·0433 (8) | 0·3560 (9) | 6·4 (3)* |
| C(65) | 0·4663 (8) | −0·0269 (8) | 0·3692 (8) | 5·9 (3)* |
| C(66) | 0·4288 (7) | −0·0841 (7) | 0·3049 (7) | 5·0 (3)* |
| C(71) | 0·3179 (6) | −0·2603 (6) | 0·1643 (6) | 3·4 (2)* |
| C(72) | 0·2444 (6) | −0·3046 (6) | 0·1326 (6) | 3·8 (2)* |
| C(73) | 0·2495 (7) | −0·3816 (7) | 0·1565 (7) | 5·2 (3)* |
| C(74) | 0·3288 (8) | −0·4113 (7) | 0·2112 (8) | 5·8 (3)* |
| C(75) | 0·4014 (8) | −0·3653 (7) | 0·2407 (8) | 5·7 (3)* |
| C(76) | 0·3980 (7) | −0·2885 (7) | 0·2179 (7) | 5·0 (3)* |
| C(81) | 0·5639 (7) | −0·1700 (6) | 0·0627 (7) | 4·0 (2)* |
| C(82) | 0·5976 (8) | −0·2418 (8) | 0·0768 (8) | 6·4 (3)* |
| C(83) | 0·629 (1) | −0·275 (1) | 0·012 (1) | 8·5 (4)* |
| C(84) | 0·624 (1) | −0·2349 (9) | −0·060 (1) | 8·0 (4)* |
| C(85) | 0·596 (1) | −0·1633 (9) | −0·074 (1) | 7·9 (4)* |
| C(86) | 0·5609 (8) | −0·1274 (8) | −0·0105 (8) | 5·9 (3)* |

$(Ph_3SbO_3SCF_3)_2O$ (II)

| | $x$ | $y$ | $z$ | $B_{eq}$, $B$ |
|---|---|---|---|---|
| Sb(1) | 0·20255 (5) | 0·0478 (1) | 0·13646 (6) | 3·07 (2) |
| Sb(2) | 0·31443 (5) | 0·3099 (1) | 0·11803 (6) | 3·29 (2) |
| S(1) | 0·0845 (3) | −0·1925 (5) | 0·1559 (3) | 5·2 (1) |
| S(2) | 0·3771 (2) | 0·6524 (5) | 0·1087 (2) | 4·5 (1) |
| F(11) | 0·0689 (7) | −0·117 (1) | 0·2760 (6) | 9·3 (4) |
| F(12) | 0·0101 (7) | −0·283 (1) | 0·2284 (7) | 10·0 (4) |
| F(13) | −0·0202 (6) | −0·097 (2) | 0·1854 (8) | 10·3 (5) |
| F(21) | 0·5018 (7) | 0·595 (2) | 0·164 (1) | 14·9 (7) |
| F(22) | 0·4779 (9) | 0·781 (2) | 0·191 (1) | 16·7 (7) |
| F(23) | 0·448 (1) | 0·621 (3) | 0·2376 (8) | 19·4 (8) |
| O(1) | 0·2846 (5) | 0·133 (1) | 0·1248 (5) | 3·4 (2)* |
| O(11) | 0·1057 (6) | −0·059 (1) | 0·1477 (6) | 4·8 (3)* |
| O(12) | 0·1394 (7) | −0·274 (2) | 0·1973 (8) | 7·9 (4)* |
| O(13) | 0·0376 (7) | −0·245 (2) | 0·0922 (8) | 7·7 (4)* |
| O(21) | 0·3573 (6) | 0·521 (1) | 0·1098 (7) | 6·3 (3)* |
| O(22) | 0·3917 (8) | 0·690 (2) | 0·0460 (8) | 8·3 (4)* |
| O(23) | 0·3333 (8) | 0·735 (2) | 0·1311 (8) | 8·3 (4)* |
| C(1) | 0·034 (1) | −0·168 (2) | 0·214 (1) | 6·2 (5)* |
| C(2) | 0·454 (1) | 0·663 (3) | 0·177 (1) | 8·7 (7)* |
| C(11) | 0·1403 (7) | 0·206 (2) | 0·1351 (8) | 3·3 (3)* |
| C(12) | 0·0875 (8) | 0·236 (2) | 0·0737 (9) | 4·1 (4)* |
| C(13) | 0·0477 (9) | 0·352 (2) | 0·0740 (9) | 4·9 (4)* |
| C(14) | 0·063 (1) | 0·430 (2) | 0·132 (1) | 6·2 (5)* |
| C(15) | 0·116 (1) | 0·400 (2) | 0·194 (1) | 5·8 (5)* |
| C(16) | 0·1571 (9) | 0·286 (2) | 0·199 (1) | 5·1 (4)* |
| C(21) | 0·2564 (8) | −0·026 (2) | 0·2371 (8) | 3·5 (3)* |
| C(22) | 0·3226 (8) | −0·074 (2) | 0·2485 (9) | 4·4 (4)* |
| C(23) | 0·3611 (9) | −0·116 (2) | 0·3183 (9) | 4·6 (4)* |
| C(24) | 0·3342 (9) | −0·104 (2) | 0·375 (1) | 5·4 (4)* |
| C(25) | 0·2682 (9) | −0·055 (2) | 0·3633 (9) | 5·1 (4)* |
| C(26) | 0·2281 (8) | −0·015 (2) | 0·2941 (8) | 3·9 (4)* |
| C(31) | 0·1813 (8) | −0·052 (2) | 0·0365 (8) | 3·7 (3)* |
| C(32) | 0·2386 (9) | −0·082 (2) | 0·0165 (9) | 4·7 (4)* |
| C(33) | 0·229 (1) | −0·138 (2) | −0·050 (1) | 5·8 (5)* |
| C(34) | 0·161 (1) | −0·155 (2) | −0·095 (1) | 6·6 (5)* |
| C(35) | 0·104 (1) | −0·127 (2) | −0·076 (1) | 6·6 (5)* |
| C(36) | 0·1159 (9) | −0·071 (2) | −0·006 (1) | 5·4 (4)* |
| C(41) | 0·4007 (8) | 0·249 (2) | 0·0887 (8) | 3·8 (4)* |
| C(42) | 0·4248 (9) | 0·325 (2) | 0·0443 (9) | 4·8 (4)* |
| C(43) | 0·481 (1) | 0·277 (2) | 0·024 (1) | 5·6 (5)* |
| C(44) | 0·508 (1) | 0·158 (2) | 0·049 (1) | 5·8 (5)* |
| C(45) | 0·482 (1) | 0·082 (2) | 0·091 (1) | 6·1 (5)* |
| C(46) | 0·4265 (9) | 0·125 (2) | 0·114 (1) | 5·1 (4)* |
| C(51) | 0·2320 (9) | 0·371 (2) | 0·0316 (9) | 4·3 (4)* |
| C(52) | 0·2100 (9) | 0·283 (2) | −0·025 (1) | 5·0 (4)* |
| C(53) | 0·152 (1) | 0·318 (2) | −0·084 (1) | 6·9 (5)* |
| C(54) | 0·121 (1) | 0·434 (2) | −0·081 (1) | 6·7 (5)* |
| C(55) | 0·140 (1) | 0·514 (2) | −0·026 (1) | 6·5 (5)* |
| C(56) | 0·198 (1) | 0·489 (2) | 0·034 (1) | 5·8 (5)* |
| C(61) | 0·3248 (8) | 0·369 (2) | 0·2239 (9) | 4·5 (4)* |
| C(62) | 0·3480 (9) | 0·275 (2) | 0·278 (1) | 5·3 (4)* |
| C(63) | 0·351 (1) | 0·305 (2) | 0·351 (1) | 6·4 (5)* |
| C(64) | 0·332 (1) | 0·427 (2) | 0·364 (1) | 6·9 (5)* |
| C(65) | 0·306 (1) | 0·518 (2) | 0·312 (1) | 7·5 (6)* |
| C(66) | 0·300 (1) | 0·491 (2) | 0·235 (1) | 5·6 (5)* |

* Isotropically refined.

$P2_1/c$ for (I) and (II); structures solved *via* Patterson function, $\Delta F$ syntheses and full-matrix least squares on $F$ with 3712 $[I > 3\sigma(I)]$ (I) and 2652 $[I > 3\sigma(I)]$ (II) reflexions; 257 (I) and 271 (II) refined parameters, isotropic temperature factors for C, O and H and anisotropic for Sb and S (I) and for Sb, S and F (II); H atoms in geometrically calculated positions (C—H 0·95 Å) and included in $F$ calculation but not refined for (I) and not included for (II); unit weights for (I) and (II), $R = 0{\cdot}044$ (I), $R = 0{\cdot}055$ (II), $S = 4{\cdot}35$ (I) and $S = 5{\cdot}93$ (II); max. $\Delta/\sigma = 0{\cdot}01$ (I) and $\Delta/\sigma = 0{\cdot}04$ (II), largest peak in final $\Delta F$ map = 0·9 (4) e $Å^{-3}$ (I) and 0·8 e $Å^{-3}$ (II); complex neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974); programs: Enraf–Nonius *SDP* (Frenz, 1981), *ORTEP* (Johnson, 1976), *POP*1 (van de Waal, 1976).

**Discussion.** The structures of the two title compounds are shown in Figs. 1 and 2. Positional parameters and equivalent values $B_{eq}$ of the anisotropic temperature factors $\beta_{ij}$ are given in Table 1 and bond lengths and angles in Table 2.* The unit cells of (I) and (II) contain four asymmetric molecules with a bent Sb—O—Sb bridge as a central part. In both compounds the atoms bound to the Sb atoms form distorted trigonal bipyramids with three C(phenyl) atoms in the equatorial plane and the bridging O and O of the unidentately bonded sulfonate ligand in apical positions. The deviations from the geometry of an ideal trigonal bipyramid around Sb are in the same range as in $(Ph_3SbO_3SCH_2CH_2OH)_2O$ (Preut, Rüther & Huber, 1985) in which the Sb—O—Sb bridge is linear. No correlation seems to exist between Sb—$\mu$-O bond lengths in comparable compounds with bent or linear Sb—O—Sb bridges: Sb—O distances in (I) [1·963 (5) Å] and (II) [1·980 (8) and 1·937 (10) Å] correspond to 1·985 (3) Å in $(Ph_3SbN_3)_2O$ with a bent bridge (Ferguson & Ridley, 1973) and also to 1·974 (2) Å in $(Ph_3SbOO\text{-}tert\text{-}C_4H_9)_2O$ (Starikova, Shchegoleva,

Table 2. *Bond lengths* (Å) *and angles* (°) *for* $(Ph_3SbO_3SC_6H_5)_2O$ (I) *and* $(Ph_3SbO_3SCF_3)_2O$ (II)

| | Compound (I) | | Compound (II) | |
|---|---|---|---|---|
| | *i*=1, *k*=1 | *i*=2, *k*=5 | *i*=1, *k*=1 | *i*=2, *k*=4 |
| Sb(*i*)—O(1) | 1·963 (5) | 1·963 (4) | 1·980 (8) | 1·937 (10) |
| Sb(*i*)—O(*i*1) | 2·247 (5) | 2·280 (5) | 2·347 (9) | 2·37 (1) |
| Sb(*i*)—C(*k*1) | 2·108 (8) | 2·11 (1) | 2·07 (1) | 2·13 (1) |
| Sb(*i*)—C(*k*+1,1) | 2·109 (9) | 2·12 (1) | 2·08 (1) | 2·08 (2) |
| Sb(*i*)—C(*k*+2,1) | 2·09 (1) | 2·10 (1) | 2·13 (2) | 2·10 (2) |
| S(*i*)—O(*i*1) | 1·503 (6) | 1·489 (6) | 1·47 (1) | 1·42 (1) |
| S(*i*)—O(*i*2) | 1·427 (7) | 1·44 (1) | 1·44 (1) | 1·41 (1) |
| S(*i*)—O(*i*3) | 1·434 (8) | 1·43 (1) | 1·42 (1) | 1·41 (1) |
| Sb(1)···Sb(2) | 3·686 (1) | | 3·639 (1) | |
| S(*i*)—C(*k*+3,1) | 1·775 (7) | 1·775 (10) | 1·79 (2)* | 1·72 (2)† |
| Sb(1)—O(1)—Sb(2) | 139·8 (3) | | 136·5 (5) | |
| O(1)—Sb(*i*)—O(*i*1) | 177·1 (2) | 172·9 (3) | 178·1 (4) | 176·6 (3) |
| O(1)—Sb(*i*)—C(*k*1) | 95·1 (3) | 93·1 (3) | 101·3 (5) | 92·9 (5) |
| O(1)—Sb(*i*)—C(*k*+1,1) | 90·1 (3) | 100·1 (3) | 92·9 (4) | 98·3 (5) |
| O(1)—Sb(*i*)—C(*k*+2,1) | 94·4 (3) | 92·6 (3) | 92·7 (4) | 98·1 (6) |
| O(*i*1)—Sb(*i*)—C(*k*1) | 87·8 (3) | 84·0 (3) | 80·2 (5) | 83·8 (5) |
| O(*i*1)—Sb(*i*)—C(*k*+1,1) | 88·3 (3) | 86·9 (3) | 87·5 (4) | 83·9 (5) |
| O(*i*1)—Sb(*i*)—C(*k*+2,1) | 83·9 (3) | 83·6 (3) | 85·6 (4) | 83·0 (6) |
| C(*k*1)—Sb(*i*)—C(*k*+1,1) | 126·6 (4) | 113·7 (4) | 116·5 (6) | 114·2 (6) |
| C(*k*1)—Sb(*i*)—C(*k*+2,1) | 119·8 (4) | 122·6 (4) | 114·8 (6) | 121·0 (5) |
| C(*k*+1,1)—Sb(*i*)—C(*k*+2,1) | 112·7 (3) | 121·3 (4) | 126·0 (6) | 121·1 (6) |
| Sb(*i*)—O(*i*1)—S(*i*) | 125·1 (4) | 142·9 (4) | 137·8 (4) | 173·1 (7) |
| O(*i*)—S(*i*)—O(*i*2) | 110·4 (4) | 109·8 (4) | 113·6 (7) | 114·8 (9) |
| O(*i*1)—S(*i*)—O(*i*3) | 110·6 (4) | 112·4 (5) | 113·7 (8) | 111·0 (8) |
| O(*i*1)—S(*i*)—C(*k*+3,1) | 104·2 (4) | 102·3 (4) | 101·0 (8)* | 104 (1)† |
| O(*i*2)—S(*i*)—O(*i*3) | 117·0 (4) | 116·8 (6) | 118·3 (9) | 116 (1) |
| O(*i*2)—S(*i*)—O(*k*+3,1) | 106·2 (4) | 106·6 (5) | 103·7 (9)* | 105·2 (9)† |
| O(*i*3)—S(*i*)—C(*k*+3,1) | 107·6 (4) | 107·9 (5) | 103·7 (8)* | 104 (1)† |

C—C distances and C—C—C angles are in the range: (I) 1·33 (2) to 1·46 (2) Å [mean value: 1·39 (2) Å] and 116 (1) to 124 (1)° [mean value: 120 (1)°]; (II) 1·31 (3) to 1·50 (3) Å [mean value: 1·40 (3) Å] and 114 (2) to 125 (2)° [mean value: 120 (2)°].

* C(1) instead of C(*k*+3,1).

† C(2) instead of C(*k*+3,1).

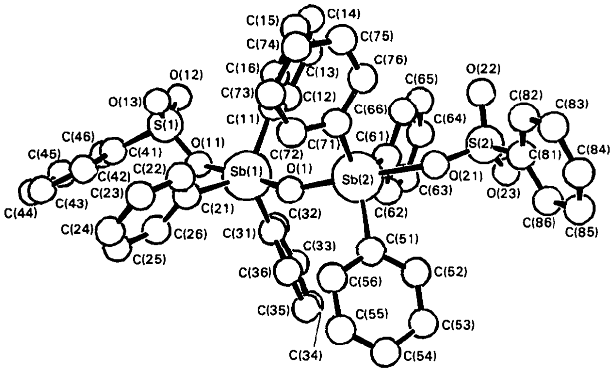

Fig. 1. General view of $(Ph_3SbO_3SC_6H_5)_2O$, (I).

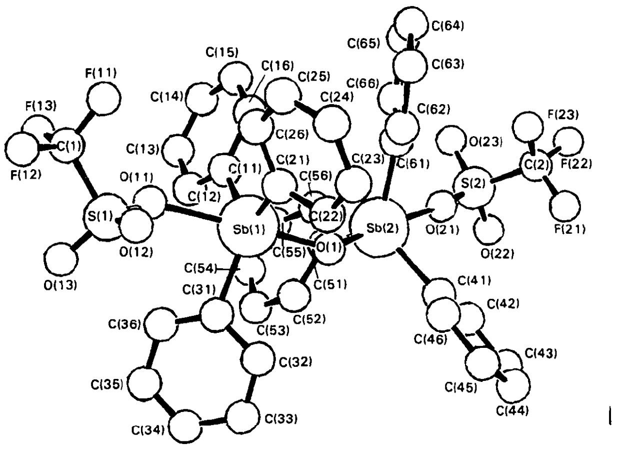

Fig. 2. General view of $(Ph_3SbO_3SCF_3)_2O$, (II).

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* Lists of structure factors, anisotropic thermal parameters and a complete list of atom parameters and C—C distances and C—C—C angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42958 (45 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Trunov & Pokrovskaya, 1978) and 1·936 (1) Å in $(Ph_3SbO_3SCH_2CH_2OH)_2O$ (Preut, Rüther & Huber, 1985) with linear bridges. We suppose that bond lengths in the Sb—O—Sb bridges are mainly influenced by the nature of the ligand *trans* to the bridging oxygen. It also appears to us that differences between the Sb—O—Sb and the Sb—O—S angles of the two title compounds and the compound $(Ph_3SbO_3SCH_2CH_2OH)_2O$ are probably caused by packing effects. Differences in Sb—O(sulfonate) distances can be related with the stronger *—I* effect of $CF_3$ and a higher ionic character can be inferred for the Sb—O(sulfonate) bond in (II) than in (I).

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# Structural Investigations of $Ni^{II}$ Complexes. VI. Two Isomers of Tetrakis(3-ethylpyridine)-bis(isothiocyanato)nickel(II)

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**Abstract.** $[Ni(NCS)_2(C_7H_9N)_4]$. Complex (I): $M_r =$ 603·6, triclinic, $P\bar{1}$, $a =$ 10·302 (3), $b =$ 9·265 (3), $c =$ 8·806 (3) Å, $\alpha =$ 108·43 (5), $\beta =$ 97·46 (5), $\gamma =$ 90·48 (5)°, $V =$ 789·6 (5) Å$^3$, $Z = 1$, $D_x =$ 1·27 g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·71069 Å, $\mu =$ 7·7 cm$^{-1}$, $F(000) = 318$, room temperature, $R =$ 0·042 for 3270 diffractions. Complex (II): $M_r =$ 603·6, monoclinic, $P2/c$, $a =$ 10·197 (3), $b =$ 12·164 (4), $c =$ 16·172 (5) Å, $\beta =$ 123·88 (5)°, $V =$ 1665 (1) Å$^3$, $Z = 2$, $D_x =$ 1·20 g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·71069 Å, $\mu =$ 7·3 cm$^{-1}$, $F(000) = 636$, room temperature, $R =$ 0·056 for 2208 diffractions. The coordination polyhedra around Ni are distorted octahedra with an $NiN_6$ coordination sphere, NCS ligands being in *trans* positions in both complexes. The *trans*-coordinated pyridine rings in complex (I) are parallel, while for complex (II) these rings are almost perpendicular.

**Introduction.** $Ni^{II}$ complexes with 3-*R*py (*R* is any bulky substituent on the pyridine ligand) form two isomers of composition $[Ni(NCS)_2(3\text{-}R\text{py})_4]$ depending on the preparation mode and the solvent properties used for the syntheses: blue (I) and violet (II) (Jamnický & Jóna,, 1982). Crystal-structure analysis of (I) and (II), $[Ni(NCS)_2(3\text{-Etpy})_4]$, showed the nature of their structural differences causing the actual phenomenon.

**Experimental.** Blue plates of (I) elongated in *z* direction [from ethanolic solution of $Ni(NCS)_2$ and 3-ethylpyridine], crystal dimensions adapted to 0·03 × 0·035 × 0·04 cm; violet prisms of (II) [from aqueous solution of $Ni(NO_3)_2$ and 3-ethylpyridine to which KSCN was added], 0·042 × 0·04 × 0·032 cm; Syntex $P2_1$ diffractometer, ten diffractions for lattice-parameter measurements with $4 \leq \theta \leq 15°$ (I) and $6 \leq \theta \leq 15°$ (II), 3270 (for I) and 2208 (for II) independent diffractions with $I > 1{\cdot}96\sigma(I)$, $2\theta \leq 55°$, $\theta$–$2\theta$ scan mode, scan speed 2·0–29·3° min$^{-1}$, *hkl* range 0, −12, −12 to 14, 12, 11 (for I) and 0, 0, −20 to 13, 15, 20 (for II), two check diffractions at intervals of 50, no significant intensity deterioration, Lp correction, absorption ignored; Patterson and Fourier methods, Et-group H atoms of complex (I) and two H of complex