# Metal Co-ordination and Hydrogen-bonding in Antimony Hydrogen Bis(thioglycollate)

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ANTIMONY HYDROGEN BIS(THIOGLYCOLLATE) was prepared by dissolving solid $Sb_2O_3$ in a hot solution of thioglycollic acid ($SH{\cdot}CH_2{\cdot}CO_2H$) in water (1/1 vol./vol.).[1] After cooling, small colourless plates of the salt were obtained. The crystals were monoclinic and the space group, at least of non-hydrogen atoms, was found to be $I2/a$ with the cell dimensions $a = 12{\cdot}414 \pm 0{\cdot}009$, $b = 12{\cdot}097 \pm 0{\cdot}007$, $c = 12{\cdot}048 \pm 0{\cdot}008$ Å and $\beta = 109{\cdot}50 \pm 0{\cdot}02°$. $D_m = 2{\cdot}45$ g.cm.$^{-3}$ and $D_c = 2{\cdot}38$ g.cm.$^{-3}$ for a cell content of eight formula units, $Sb(SCH_2{\cdot}CO_2)_2H$.

The crystal structure was solved from 1017 independent reflections, registered in a Weissenberg camera with Cu-$K_\alpha$ radiation and refined by least-squares technique to a final discrepancy factor $R = 12{\cdot}1\%$. The compound was found to be a chelate, the organic part forming two five-membered rings with the antimony atom.

The antimony atom is co-ordinated to two oxygen and two sulphur atoms in a one-sided four-fold arrangement with bond angles $\alpha = 157{\cdot}6°$, $\beta = 88{\cdot}1°$, $\gamma = 79{\cdot}1°$, $\delta = 80{\cdot}9°$, $\epsilon = 82{\cdot}7°$, $\zeta = 97{\cdot}5°$ (*cf.* Figure 1). The co-ordination can be described as a distorted trigonal bipyramid, where two equatorial apices are occupied by sulphur atoms (Sb–S distances of $2{\cdot}43 \pm 0{\cdot}01$ Å) and the two axial apices by oxygen atoms (Sb–O distances $2{\cdot}30 \pm 0{\cdot}02$ and $2{\cdot}26 \pm 0{\cdot}02$ Å) while the fifth apex corresponds to an unshared electron pair. Such an arrangement around antimony was first suggested by Edstrand[2] and later found in a number of antimony compounds, *e.g.*, $\beta$-$Sb_2O_4$.[3]

Two very short intermolecular oxygen–oxygen distances ($2{\cdot}43 \pm 0{\cdot}04$ and $2{\cdot}54 \pm 0{\cdot}04$ Å) suggest the presence of symmetrical hydrogen bonds. These bonds link the molecules to form endless chains (*cf.*, Figure 2). Hydrogen bonds of about 2·4 Å observed in several compounds have been reported to be truly or statistically symmetrical, *e.g.*, the O–H–O distance of 2·44 Å in potassium hydrogen maleate.[4] Even distances as long as 2·5—2·6 Å may be symmetrical *e.g.*, the O–H–O distance of 2·54 Å in potassium hydrogen diphenylacetate.[5]

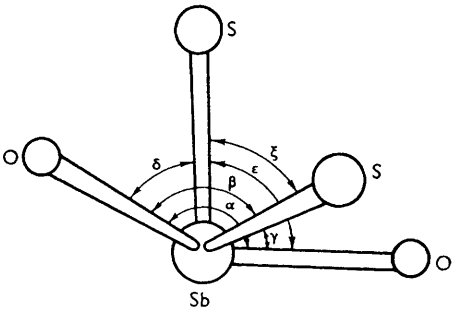


FIGURE 1. *The co-ordination around an antimony atom.*

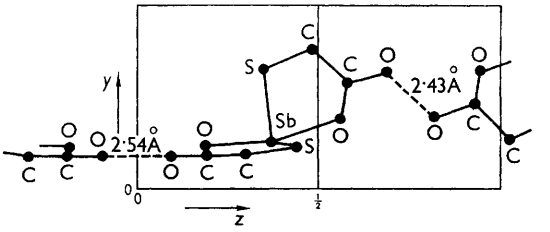


FIGURE 2. *Projection along the* a*-axis showing the hydrogen bonds.*

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