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The crystal structure of trimethyltin hydroxide

Recently, there has been much interest in organotin compounds in which the tin atoms are penta- or hexa-coordinated. Though the spectroscopic studies on the structure of trialkyltin acetate[1], formate[2], perchlorate[3,4], nitrate[5], or trimethyltin hydroxide[6,7] suggested that the trialkyltin group in these compounds is weakly coordinated by two oxygen atoms to give a penta-coordinated tin atom, no X-ray diffraction study of these compounds has been reported. Therefore, it seemed worthwhile to determine the crystal structure of trimethyltin hydroxide, which is one of the simplest oxygen-containing organotin compounds.

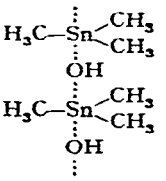
Crystals of trimethyltin hydroxide, $(CH_3)_3SnOH$, are monoclinic. The dimensions of the unit cell are given in Table 1. The mean intensity of (*hkl*) reflections is

TABLE 1

THE UNIT CELL OF TRIMETHYLTIN HYDROXIDE

True cell	*Sub-cell 1*	*Sub-cell 2*
$a = 13.34$ Å	$a' = 6.67$ Å	$a' = 6.67$ Å
$b = 33.20$	$b = 33.20$	$b' = 4.15$
$c = 22.42$	$c' = 11.21$	$c' = 11.21$
$\beta = 90°$	$\beta = 90°$	
$Z = 64$	$Z = 16$	$Z = 2$
Pn ?	*Pn*	$Pn2_1m$

strong when $k = 8n$, weak when $k = 8n \pm 3$, and not observed when $k = 8n \pm 1$, $8n \pm 2$ or $8n \pm 4$. This feature of the intensity of the layer lines suggests the presence of an 8_3 helix-like molecular chain in the crystal; moreover, the reflections in these observed layer lines are very weak when h or l is odd. For this reason the analysis was first made on the sub-cell 2 (Table 1). The structure was determined by Patterson methods, and refined by three-dimensional Fourier syntheses. R is 0.11 for 232 observed reflections. The structure consists of chains of trimethyltin groups and hydroxyl groups arranged alternately along the b' axis, and the chains are held together with weak Van der Waals forces between the methyl groups in the neighboring chains. The oxygen atom lies on the axis of, and is almost equidistant between, two tin atoms. No hydrogen bond is found in this crystal. The trimethyltin group is nearly planar, which is consistent with the observed infrared spectrum[6,7], and inclined at about 15° with respect to the plane perpendicular to the chain axis.



This result was extended to the sub-cell 1, in which the length of the b axis is eight times of that of the sub-cell 2. In each molecular chain, the 8_3 helical arrangement of tin atoms along the chain axis was first assumed. The refinement by trial and error method showed that in the sub-cell 1 the tin atoms are arranged to form a distorted 8_3 helix. The projection of the helix perpendicular to the chain axis gives an ellipse with the major axis length 0.20 Å along the c axis and minor axis 0.10 Å along the a axis. R is 0.12 for 401 observed reflections.

The extention to the true cell ($a = 2a'$, $c = 2c'$) should take into account of another ten very weak reflections. These reflections suggest that there may be some disorder in the chain arrangement along the a and c axes.

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