988. *The Crystal and Molecular Structure of Tetrachloro-*1,4-*bistriethylstannyloxybenzene.*

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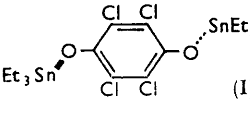
The crystal structure of tetrachloro-1,4-bistriethylstannyloxybenzene has been determined by *X*-ray diffraction methods. The molecules exist in the *trans*-conformation. The Sn–O distance is 2·08 Å.

DURING an investigation of some organotin compounds, we wished to know the conformation of tetrachloro-1,4-bistriethylstannyloxybenzene (I). Models show that free rotation round the C–O bonds is precluded by the bulky chlorine and ethyl groups. The molecules must therefore, be constrained to the *cis*- or *trans*-form. A two-dimensional *X*-ray crystal analysis was carried out to ascertain the molecular geometry, also to determine a value for the Sn–O covalent distance, a value not hitherto recorded.



(I)

Experimental.—$C_{18}H_{30}Cl_4O_2Sn_2$. $M = 657{\cdot}6$. Monoclinic. $a = 8{\cdot}72 \pm 0{\cdot}02$, $b = 11{\cdot}59 \pm 0{\cdot}03$, $c = 12{\cdot}96 \pm 0{\cdot}03$ Å, $\beta = 97{\cdot}9° \pm 0{\cdot}2°$. $U = 1297$ Å^3. $D_m = 1{\cdot}64$ (by flotation), $Z = 2$, $D_c = 1{\cdot}683$, $F(000) = 576$. Space group, $P2_1/n$ (C^5_{2h}, No. 14). Cu-K_α radiation ($\lambda = 1{\cdot}542$ Å), single crystal rotation and Weissenberg photographs.

Multiple-film Weissenberg photographs were taken round [*a*] and [*b*]. Very small crystals were used in order to minimize absorption ($\mu = 197$ cm.$^{-1}$). Relative intensities were estimated visually by comparison with standard strips prepared from the same crystals. 103 0*kl* and 96 *h*0*l* reflexions were observed to be non-zero. Each projection was solved from Patterson syntheses, which served to locate the tin and the chlorine atoms. The lighter atoms were then found from Fourier maps and with the assistance of known lengths and angles. The structure was refined so far as possible by difference syntheses. The final agreement index was $R = 7{\cdot}5\%$ for the 0*kl* and $R = 7{\cdot}0\%$ for the *h*0*l* projection. These figures include only the observed reflexions. The scattering factors used were those of Berghuis *et al.*[1] for carbon and oxygen, that of Tomiie and Stam [2] for chlorine, and that of Thomas and Umeda [3] for the tin atom. Thomas and Umeda's figures were corrected for the real part of the dispersion by means of the Table given by Dauben and Templeton.[4] Isotropic temperatures factors $B = 7{\cdot}9$ Å^2 and $5{\cdot}7$ Å^2 were required for the 0*kl* and *h*0*l* projections, respectively.

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[1] Berghuis, Haanappel, Potters, Loopstra, MacGillavry, and Veenendaal, *Acta Cryst.*, 1955, **8**, 478.
[2] Tomiie and Stam, *Acta Cryst.*, 1958, **11**, 126.
[3] Thomas and Umeda, *J. Chem. Phys.*, 1957, **26**, 293.
[4] Dauben and Templeton, *Acta Cryst.*, 1955, **8**, 841.

TABLE 1.

Fractional co-ordinates.

Atom	*x/a*	*y/b*	*z/c*
Sn	0·118	0·767	0·2675
Cl$_1$	0·345	0·988	0·082
Cl$_2$	0·757	0·924	0·146
O	0·100	0·933	0·207
C$_1$	0·157	0·000	0·028
C$_2$	0·060	0·962	0·110
C$_3$	0·890	0·962	0·067
C$_4$	0·032	0·728	0·411
C$_5$	0·942	0·833	0·445
C$_6$	0·368	0·755	0·283
C$_7$	0·442	0·857	0·350
C$_8$	0·978	0·643	0·165
C$_9$	0·803	0·617	0·167

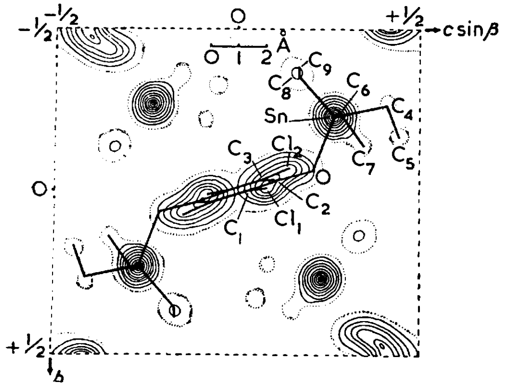

FIG. 1. (a) Projection of the contents of the unit cell down [*a*].

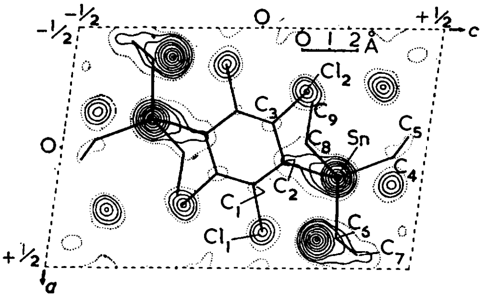

FIG. 1. (b) Projection of the contents of the unit cell down [*b*].

(The contours are drawn at equal, arbitrary intervals, except in the tin peaks, where alternate contours are omitted.)

TABLE 2.

Observed and calculated structure factors for one asymmetric unit.

0kl	*F*$_o$	*F*$_c$
0.0.2	42·7	−42·0
4	12·2	13·3
6	22·0	−20·8
8	6·7	6·4
10	5·4	−5·6
0.1.1	28·0	29·4
2	4·9	5·4
3	2·6	−2·4
4	7·5	−6·2
5	8·9	−9·8
6	17·8	17·7
7	2·4	−3·0
8	13·4	−14·7
10	8·4	8·4
12	5·4	−4·5
14	2·7	2·2
0.2.0	15·6	−16·1
1	21·2	−22·8
2	47·2	47·1
3	1·5	−0·8
4	36·7	−37·7
5	5·7	−4·2
6	13·8	14·7
7	3·0	3·4
8	10·2	−9·9
10	4·4	3·8
0.3.1	30·0	29·3
2	32·5	−33·5
3	9·6	−8·9
4	16·2	17·2
6	10·3	−11·7
7	7·0	−6·6
8	9·2	10·1
9	1·5	0·5
10	7·6	−6·5
11	1·9	−1·2
12	4·4	4·9
0.4.0	36·3	37·3
1	1·7	−1·5
2	18·3	−16·4
3	15·6	−16·4
4	21·4	22·9
0.4.5	10·4	11·6
6	24·5	−23·6
7	3·6	−3·2
8	5·2	5·2
9	2·1	2·1
0.5.1	4·8	6·0
2	4·4	−5·2
3	8·5	8·1
4	9·9	−10·3
5	10·2	−9·2
6	10·5	9·1
7	1·2	0·5
8	6·4	−7·1
9	3·8	−3·4
10	4·9	4·4
11	3·6	4·1
12	1·5	−1·8
0.6.0	16·2	−17·1
1	19·5	−19·3
2	22·9	24·6
3	7·4	7·6
4	11·0	−11·1
5	6·7	−7·1
6	3·7	3·8
7	3·5	3·9
8	5·1	−6·1
10	2·7	2·3
0.7.1	1·5	−0·5
2	5·8	−5·1
5	2·0	1·9
6	4·1	−4·8
7	6·0	−7·1
8	6·4	6·3
9	3·8	3·8
0.8.0	9·4	9·9
1	10·4	8·7
2	5·2	−5·6
3	11·1	−11·7
4	5·0	6·2
5	3·3	2·8
6	4·9	−5·4
7	3·4	−2·4
0.8.8	1·2	1·2
9	2·4	3·0
0.9.3	4·1	4·6
4	4·6	−4·7
5	4·1	−4·4
7	1·4	1·4
9	2·7	−2·2
0.11.5	2·1	1·8
7	1·9	−1·0
h0l		
0.0.2	42·1	−42·6
4	12·2	14·0
6	25·0	−23·4
8	9·2	7·9
10	6·9	−7·9
1.0.1	32·1	−30·5
3	19·1	17·8
5	34·3	−32·7
7	29·2	30·3
9	7·9	−7·3
11	5·2	4·1
13	5·9	−6·4
2.0.2	10·9	11·9
4	5·5	3·9
6	4·7	3·8
8	16·9	−15·3
10	6·8	5·8
12	6·9	−7·2
14	3·4	3·7
3.0.1	41·5	−42·3
3	14·3	13·3
5	7·4	−6·4
7	2·9	2·4
9	2·3	−1·3
13	3·1	3·4
4.0.2	11·5	12·2
4	26·8	−27·5
6	20·2	22·1
10	3·0	2·4
12	1·6	−1·1
5.0.1	17·2	18·4
3	9·6	−10·3
5	12·6	14·0
7	6·2	−5·2
5.0.9	6·2	5·9
11	4·9	−5·8
13	2·5	2·1
6.0.6	5·9	−6·0
8	4·2	2·8
12	2·4	3·3
7.0.3	8·6	−9·1
5	6·4	7·1
8.0.2	5·8	−6·9
4	5·6	6·5
6	3·2	−3·7
9.0.5	4·1	−4·8
$\bar{1}$.0.1	33·1	30·1
3	32·9	−31·3
5	32·7	33·7
9	10·5	−10·4
11	5·4	5·7
13	3·7	−3·4
15	2·9	2·9
$\bar{2}$.0.2	14·3	−15·5
4	9·4	8·5
6	17·4	−17·5
8	14·9	15·1
10	3·6	−2·3
12	9·0	8·0
14	7·0	−7·3
16	2·4	2·3
$\bar{3}$.0.1	48·1	49·8
3	32·3	−30·7
5	8·5	7·3
7	21·0	−22·5
9	15·4	15·2
11	4·9	−5·3
13	4·0	4·8
$\bar{4}$.0.2	32·5	32·8
4	28·5	−28·9
6	20·3	21·3
8	4·9	−4·0
$\bar{5}$.0.1	18·3	−18·4
3	2·7	−1·2
13	4·7	4·0
15	3·5	−4·2
$\bar{6}$.0.2	12·2	11·3
6	5·2	5·1
$\bar{6}$.0.8	12·3	−11·9
10	7·6	8·5
12	3·4	−3·1
$\bar{7}$.0.1	11·1	−11·7
3	20·0	19·6
5	9·3	−8·2
7	7·7	6·8
9	3·7	−4·7
$\bar{8}$.0.2	9·3	−9·7
6	3·8	−3·3
8	3·9	4·7
$\bar{9}$.0.9	3·8	−3·8
$\overline{10}$.0.2	3·0	− 4·0
4	4·1	4·4

Results.—The co-ordinates of the atoms are given in Table 1. Figs. 1a and b show the final Fourier maps for the $0kl$ and $h0l$ projections respectively. Fig. 2 shows the bond lengths and the numbering of the atoms. The C–Cl bonds are estimated to be accurate to ±0·05 Å, the Sn–O and Sn–C bonds to ±0·06 Å, and the bonds between light atoms to ±0·1 Å. Table 2 gives the observed and the calculated structure factors.

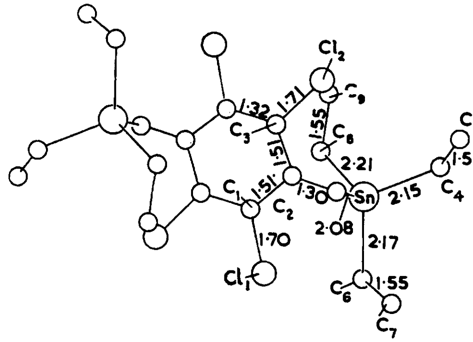

FIG. 2. The numbering of the atoms, and the bond lengths.

The molecules possess centres of symmetry and thus all exist in the *trans*-form in the crystal. No detectable changes were found in the angles formed by the tin atom, which were assumed to be tetrahedral. The plane containing the atoms, Sn, O, and C_2 is normal to the plane of the quinone ring. The Sn–O distance is 2·08 Å, close to the sum of Pauling's covalent radii.[5] The angle at the oxygen is 127°. Other bond lengths, bond angles, and intermolecular distances have normal values.

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[5] Pauling, " The Nature of the Chemical Bond," Cornell Univ. Press, 1960, p. 164.