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**Preliminary communication**

# THE X-RAY STRUCTURE OF 2,2-DIBUTYL-1,3,2-DIOXASTANNOLANE

ALWYN G. DAVIES, ALLAN J. PRICE
*Chemistry Department, University College London, 20 Gordon Street, London WC1H 0AJ (Great Britain)*
HELEN M. DAWES and MICHAEL B. HURSTHOUSE
*Chemistry Department, Queen Mary College, Mile End Road, London E1 4NS (Great Britain)*

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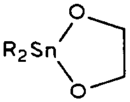
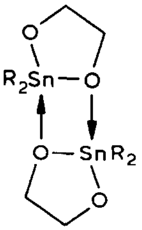
## Summary

In the crystal, 2,2-dibutyl-1,3,2-dioxastannolane exists as an infinite ribbon coordination polymer, containing six-coordinate tin at highly distorted octahedral sites.

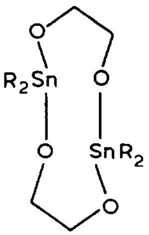
The 2,2-dialkyl-1,3,2-dioxastannolanes (I) are finding increasing use in organic synthesis because they confer regiospecific activation on 1,2-diols [1], and they can react with diacyl chlorides or diisocyanates to form tetralactones or tetraurethanes [2]. Molecular weight measurements show that the compounds I usually exist in solution as dimers [3—6], although there is evidence for the presence of monomers or oligomers under certain conditions, and the above reactions have been interpreted in terms of the dimer structures II or III.



(I)



(II)



(III)

A number of unpublished attempts have been made to determine the structures of these compounds by single crystal X-ray diffraction, but none has been successful for derivatives of simple diols.

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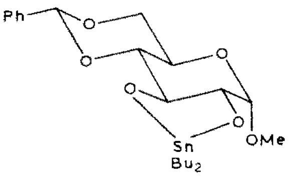
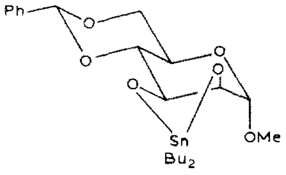
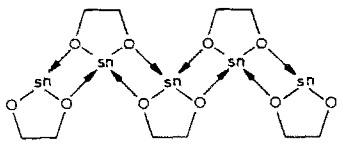


(IV)



(V)



(VI, sn = Bu$_2$Sn)

The dibutylstannylene derivatives of monosaccharides have proved more tractable, and the structures of two such compounds have been published. Methyl 4,6-di-*O*-benzylidene-2,3-*O*-dibutylstannylene-α-D-glucopyranoside (IV) has been shown to exist as a coordination dimer with 5-coordinate tin atoms (cf. II) [7], but the corresponding mannose derivative V in the crystal has the structure of a coordination pentamer in which the tin atoms are 5-coordinate in the two terminal units and 6-coordinate in the three medial units (cf. VI) [8].

We now report the determination of the structure of 2,2-dibutyl-1,3,2-dioxastannolane itself (I, R = Bu).

A view of a section of the molecule is shown in Fig. 1*. It is an infinite

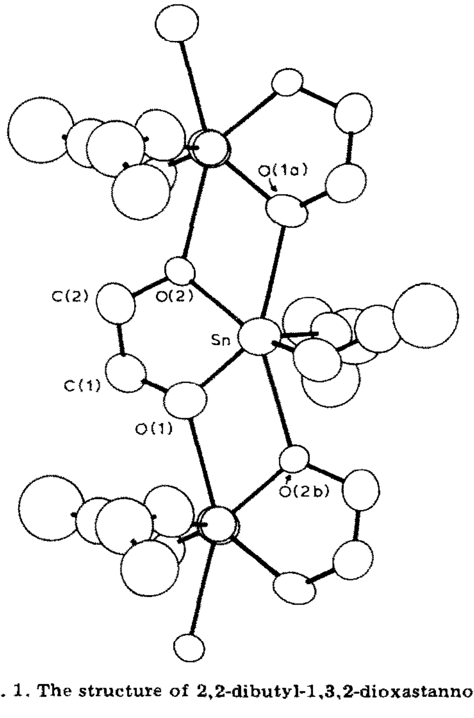


Fig. 1. The structure of 2,2-dibutyl-1,3,2-dioxastannolane.

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*[*Crystal data*: $C_{10}H_{22}O_2Sn$, $M = 293$, orthorhombic, space group $P2_1\,2_1\,2_1$, $a$ 8.103(4), $b$ 7.409(3), $c$ 21.563(4) Å, $Z = 4$, $D_C$ 1.39 g cm$^{-3}$, $\mu$-(Mo-$K_\alpha$) 17.93 cm$^{-1}$, 1011 observed out of 1346 measured reflections [$I > 1.5\,\sigma(I)$], data measured to $\sin\theta/\lambda = 0.595$ Å$^{-1}$, $R = 0.069$ (anisotropic thermal parameters for all non-hydrogen atoms, high thermal motion for atoms of the butyl groups; hydrogen atoms could not be located). The atomic coordinates for this work are available on request from the Director of the Cambridge Crystallographic Data Centre, University Chemical Laboratory, Lensfield Road, Cambridge CB2 1EW. Any request should be accompanied by the full literature citation for this communication.

ribbon coordination polymer, with 6-coordinate tin, similar to the dibutylstannylene derivative of propane-1,3-diol [9]. The polyhedron about the tin is a highly distorted octahedron with bond lengths Sn—C 2.128(24) and 2.136(22) Å to the butyl groups, Sn—O 1.975(14) and 2.097(10) Å to the oxygens O(1) and O(2) of the "chelating" diol, and Sn—O 2.495(15) and 2.520(11) Å to the oxygens O(1a) and O(2b) of two neighbouring units to complete a polymeric chain along the $2_1$ screw axis. The endocyclic angle O(1)—Sn—O(2) is 79.0(5)°, and the exocyclic angle C(3)—C(7) is 138.6(6)°.

As pointed out in ref. 8, self-association of the glucose derivative IV is limited to dimerisation because of steric hindrance by the sugar moieties which are coplanar with the dioxastannolane moiety; in the mannose derivative V, this steric hindrance is less because the *cis* bonding to the pyranose ring causes these mean planes to be orthogonal, and formation of a pentamer is permitted. In the glycol derivative, such steric hindrance is absent, and there is no restriction to the formation of an infinite polymer.

*Note added in proof.* In the light of this work, recent results on the Mössbauer spectra of the dioxastannolanes needs to be reassessed (R.H. Herber, A. Shanzer, and J. Libman, Organometallics, 3 (1984) 586).

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