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Short Communication

Crystal structure of C_2O_4 ($SnPh_3$·dimethylformamide)$_2$

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Abstract

The structure of $C_2O_4(SnPh_3)_2$. 2dimethylformamide (DMF) has been determined by X-ray. It consists of monomers containing $SnPh_3$ residue trans coordinated by DMF, with the environment around the tin centre being trigonal bipyramidal.

Keywords: bidentate oxalate; tin (IV) in a trigonal bipyramidal environment; X-ray crystallography.

The applications found for many molecules belonging to the organotin (IV) family (Evans and Karpel, 1985) explains our interest in the synthesis of new tin (IV) containing molecules. The study of the interactions between C_2O_4 $(SnPh_3)_2$ and dimethylformamide has led to the studied adduct. We have previously published the structure of $C_2O_4(SnPh_3)_2$ (Diop et al., 2003) and $SeO_3(SnPh_3)_2.CH_3OH$ (Diallo et al., 2007) in the dynamic of our interest on the coordinating behaviour of the oxyanions.

The asymmetric unit of the adduct $C_2O_4(SnPh_3$.dimethylformamide [DMF])$_2$ half of the molecule, with the remaining portion being generated by an inversion centre at the midpoint of the C(1)-C(1′) bond. The DMF located at the axial site of a trigonal bipyramid coordinates to tin through its oxygen atom [Sn(O1)-O(3) 2.4159(17) Å]-in $(SnPh_3)_2VO_4$.2DMF (Herntrich and Merzweiller, 2006) the SnO bond is 2.466 Å. The sum of the C-Sn-C angles (358.1°) reveals an almost planar SnC_3 group. The O-Sn-O moiety deviates a little from linearity as evidenced by the value of O(1)-Sn-O(3) angle [177.88(6)°]. The oxalate group is monodentate with respect to Sn(1) but acts as a bridging group between neigbouring tin centres. As a result, the bond lengths of C(1)-O(1) [1.299(3) Å] and C(1)-O(2) [1.221(3) Å] indicate, respectively, are of single and double bond character. There are weak hydrogen bonds between the oxalate oxygens and two hydrogens of one methyl group of DMF [H21A..O2 2.791; H21C..O1 3.031 Å], which generates a supramolecular array.

The structure of the title compound is discrete, the environment of the tin centre being trigonal bipyramidal with almost co-planar equatorial positions.

Experimental

When $C_2O_4(SnPh_3)_2$ obtained as per Diop et al. (2003) and is dissolved in dimethylformamide, crystals of C_2O_4 ($SnPh_3$·dimethylformamide)$_2$ that are suitable for X-ray work are obtained after a slow solvent evaporation. All the chemicals were purchased from Aldrich (Germany) and used as such.

A crystal of approximate dimension of 0.2×0.2×0.2 mm was used for data collection. Data were collected at 150(2) K using Mo-k_α radiation (λ=0.71073 Å); refinement was full-matrix least-squares based on F^2 and the absorption correction was semi-empirical from equivalents. In the final cycles of least-squares refinement, all non-hydrogen atoms were allowed to vibrate anisotropically.

Table 1 Crystal data of the title compound.

Empirical formula	C44 H44 N2 O6 Sn_2
Formula weight	934.19
Temperature	150(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	P 1 2_1/n 1
Unit cell dimensions	a=8.8397(2)Å α=90°
	b=16.1375(4) Å β=105.5600(10)°
	C=14.9160(3) Å γ=90°
Volume	2049.79(8) $Å^3$
Z	2
Density (calculated)	1.514 Mg/m^3
Absorption coefficient	1.267 mm^{-1}
F(000)	940
Crystal size	0.20×0.20×0.20 mm
θ range for data collection	3.99–30.04°.
Index ranges	-12≤h≤12; -22≤k≤22; -18≤l≤20
Reflections collected	43205
Independent reflections	5962 [R(int)=0.0674]
Reflections observed (>2σ)	4182
Data Completeness	0.995
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7857 and 0.7857
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	5962 / 0 / 246
Goodness-of-fit on F at 2	1.024
Final R indices [I>2σ(I)]	R~1=0.0336 wR~2=0.0649
R indices (all data)	R~1=0.0685 wR~2=0.0740
Largest diff. peak and hole	0.885 and -1.022 e. $Å^{-3}$

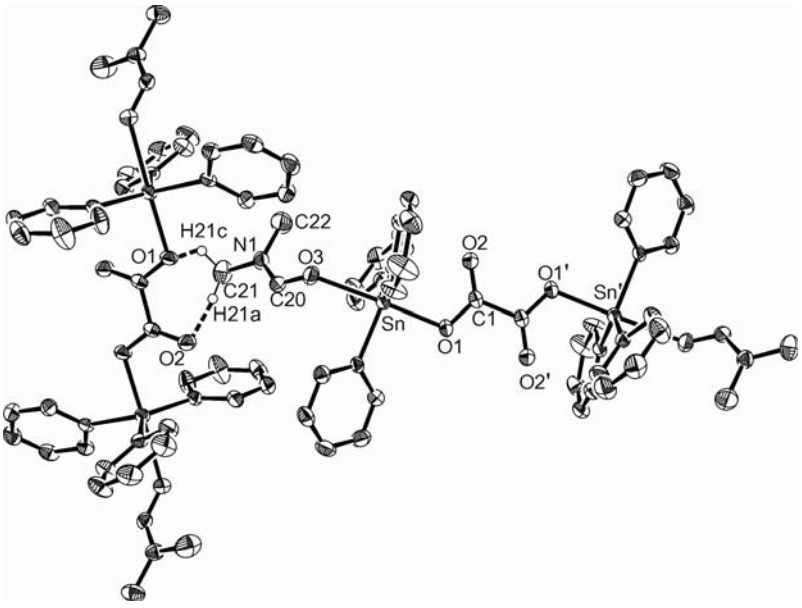

Figure 1 Schematic structural view of the title compound.
Selected bonds: Sn-O(1) 2.1412(16); Sn-O(3) 2.4159(17); O(1)-C(1) 1.299(3); O(2)-C(1) 1.221(3) Å and angles: O(1)-Sn-O(3) 177.88(6); C(14)-Sn-C(8) 114.89(9); C(14)-Sn-C(2) 129.58(9); C(8)-Sn-C(2) 113.63(9); C(14)-Sn-O(1) 95.86(8); C(8)-Sn-O(1) 90.53(7); C(2)-Sn-O(1) 96.56(8); C(14)-Sn-O(3) 84.42(7); C(8)-Sn-O(3) 87.44(7); C(2)-Sn-O(3) 84.85(8) °. (Symmetry transformation 1-x, -y, -z.).

Hydrogen atoms were included at calculated positions, when relevant. The structure has been solved by SHELXS (Sheldrick, 1986) and refined by SHELXL (Sheldrick, 1997). Crystal data are given (Table 1) and a schematic view is also shown (Figure 1).

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