Supramolecular organotin tris-carboxylates: crystal and molecular structure of $[\text{Cy}_2\text{NH}_2]_2[1-\text{Me}_3\text{(H}_2\text{O})\text{SnOCO-3,5-(OOC)}_2\text{C}_6\text{H}_3\text{]}\cdot\text{EtOH}$

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Abstract

The crystal and molecular structure of $[\text{Cy}_2\text{NH}_2]_2[\text{C}_6\text{H}_3(\text{CO}_2\text{)}_3\text{SnMe}_3\cdot\text{H}_2\text{O}]\cdot\text{CH}_3\text{CH}_2\text{OH}$ (I) has been determined by single crystal X-ray diffraction analysis. In this compound, the tin atom is pentacoordinated by three methyl groups, one oxygen atom of a water molecule and one oxygen atom deriving from the tris-carboxylato ligand, in a trans-coordinated $\text{O}_2\text{SnC}_3$ environment. The dicyclohexylammonium cations $[\text{O(3)}, \text{O(6)}]$ are involved in stronger hydrogen bonds with the non-coordinated oxygen atoms of the carboxylate, while, along with hydrogen bonds involving water and ethanol, gives rise to a complex 3D lattice structure.

Keywords: carboxylate; organotin; supramolecular; X-ray.

Introduction

Resulting from the various applications of organotin(IV) compounds (Ayre and Poller, 1980; Evans and Karpel, 1985; Alvarez Boo et al., 2006), our groups have been involved in seeking new organo- and halotin(IV) molecules and have reported several papers in this area (Diassé-Sarr et al., 1997, 2004; Diop et al., 2003; Diallo et al., 2009). We have initiated a study of the reactions between salts of a tricarboxylic acid $[\text{C}_6\text{H}_3(\text{CO}_2\text{)}_3\text{(Cy}_2\text{NH}_2\text{)}]$ and $\text{SnMe}_3\text{Cl}$ to explore the reactions between salts of a tri-carboxylic acid $[\text{C}_6\text{H}_3(\text{CO}_2\text{)}_3\text{(Cy}_2\text{NH}_2\text{)}]$ and $\text{SnMe}_3\text{Cl}$ to explore the various applications of organotin(IV) compounds (Ayrey and Poller, 1980; Evans and Karpel, 1985; Diallo et al., 2009). We have initiated a study of the reactions between salts of a tri-carboxylic acid $[\text{C}_6\text{H}_3(\text{CO}_2\text{)}_3\text{(Cy}_2\text{NH}_2\text{)}]$ and $\text{SnMe}_3\text{Cl}$ to explore the application of organotin(IV) molecules and have reported several papers in this area (Diassé-Sarr et al., 1997, 2004; Diop et al., 2003; Diallo et al., 2009).

Results and discussion

The structure (Figure 1) consists of a central benzene-1,3,5-tricarboxylato tri-anion monocoordinating a single $\text{SnMe}_3$ residue, the metal linked to a water molecule in axial position generating a trans-trigonal bipyramidal $\text{O}_2\text{SnC}_3$ environment; charge balance is achieved through the presence of two $[\text{Cy}_2\text{NH}_2]^+$ cations. The carboxylate bonded to tin is monodentate, reflected in two markedly different C–O bond lengths $[\text{C(1)}–\text{O(1)} 1.294(2), \text{C(1)}–\text{O(2)} 1.230(2) \text{Å}]$ and an $\text{Sn(1)}–\text{O(2)}$ separation of $>3.2 \text{Å}$. There is less disparity in the C–O bonds of the two remaining carboxylates [e.g., C(8)–O(5) 1.267(2), C(8)–O(6) 1.246(2) Å], both of which are involved in N–H…O hydrogen bonds.

Experimental

$[\text{C}_6\text{H}_3(\text{CO}_2\text{)}_3\text{(Cy}_2\text{NH}_2\text{)}]$ is obtained on neutralizing benzene-1,2,3-tricarboxylic acid with $\text{Cy}_2\text{NH}$ in water in 1:3 ratio; a white powder is collected after a solvent evaporation at 60°C. When an aqueous solution of $[\text{C}_6\text{H}_3(\text{CO}_2\text{)}_3\text{(Cy}_2\text{NH}_2\text{)}]$ is mixed with ethanolic solution of $\text{SnMe}_3\text{Cl}$ in 1:1 ratio, a clear solution is obtained and stirred for no less than 2 h. When submitted to a slow solvent evaporation, this solution yields crystals suitable for an X-ray study. All the chemicals were purchased from Aldrich (Germany) and used without any further purification.

Crystal structure

A crystal of approximate dimensions $0.25 \times 0.25 \times 0.20 \text{mm}$ was used for data collection. Data were collected at 150(2) K using
Mo-$\text{k}\alpha$ radiation ($\lambda=0.71073$ Å); full details of the crystallographic experiment are given in Table 1. Refinement was full-matrix least-squares based on $F^2$, 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. Hydrogen atoms when included at calculated positions were relevant, except for those of the NH$_2$, EtOH and H$_2$O groups which were located in the difference map and refined. The structure was solved and refined using the WinGX-1.70 suite of programs (Farrugia, 1999).
References


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