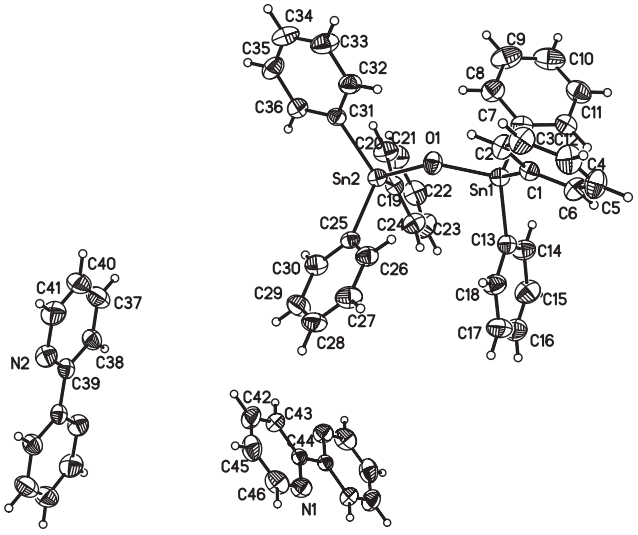
Junshan Sun* and Yan Kong

The crystal structure of 1,1,1,3,3,3-hexaphenyldistannoxane – 2,2′-bipyridine (1/1), $C_{46}H_{38}N_2OSn_2$



https://doi.org/10.1515/ncrs-2018-0457
Received October 28, 2018; accepted December 29, 2018; available online February 4, 2019

Abstract

$C_{46}H_{38}N_2OSn_2$, triclinic, $P\bar{1}$ (no. 2), $a = 9.657(4)$ Å, $b = 9.679(4)$ Å, $c = 24.324(9)$ Å, $\alpha = 95.397(5)°$, $\beta = 90.138(5)°$, $\gamma = 119.283(5)°$, $V = 1971.2(12)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0369$, $wR_{ref}(F^2) = 0.0873$, $T = 298(2)$ K.

CCDC no.: 1887913

The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

In a typical experiment, 2,3,4,5-tetrafluorobenzoic acid (0.194 g; 1 mmol), triphenyltin chloride (0.385 g, 1 mmol) and sodium ethoxide (0.748 g, 1.1 mmol), in the presence of 2,2′-bipy (0.156 g, 1 mmol) were dissolved in benzene. This mixture was heated to 40 °C for 3 h, resulting in a colorless solution. Crystals of the title compound were obtained by slow evaporation within one week.

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.33 × 0.29 × 0.27 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.30 mm^{-1}
Diffractometer, scan mode:	Bruker SMART APEX II CCD,
θ_{max}, completeness:	25.0°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	10366, 6869, 0.030
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4869
$N(param)_{refined}$:	460
Programs:	Bruker [1], SHELX [2]

Experimental details

All hydrogen atom positions were taken from a difference Fourier map. Hydrogen atoms were assigned with common isotropic displacement factors $U_{iso}(H) = 1.2$ times U_{eq}. All the H atoms were refined as riding on their parent atom.

Comment

Organotin compounds form an important class of compounds and have received much interest in recent years, not only due to their intrinsic interest but owing to their varied applications. Some are used as catalysts and stabilizers, and certain derivatives are biocides, act as antifouling agents and wood preservatives [3]. In order to explore the impact of organotin compound they have been studied intensively [4–7]. Interestingly, Gielen *et al*. reported that fluorinated organotin carboxylates own anticances activity [8, 9].

So our interest in the structures of organotin(IV) complexes containing N and O atoms has prompted us to embark the 2,3,4,5-tetrafluorobenzoate ligand and 2,2′-bipy. Unfortunately, the carboxylate did not coordinate to tin. Two triphenyltin link one oxygen atom, which is similiar to reported structures [10–13]. The crytal structure of the title complex is shown in the figure. The title complex shows additionally two 2,2′-bipy molecules both located around inversion centers. The distance of O—Sn are 1.964(3) Å and 1.967(4) Å, which

***Corresponding author: Junshan Sun,** College of Chemistry and Chemical Engineering, Taishan University, Tai'an 271000, Shandong Province, China, e-mail: sunjunshan79@tsu.edu.cn
Yan Kong: Middle School of Taishan College, Tai'an 271000, Shandong Province, China

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Sn1	0.80203(4)	0.47262(3)	0.17784(2)	0.04559(11)
Sn2	0.89118(4)	0.58633(3)	0.32355(2)	0.04600(11)
N1	−0.1008(5)	0.7895(5)	0.50785(17)	0.0689(12)
N2	0.6154(5)	0.3999(5)	0.99172(18)	0.0688(12)
O1	0.8850(4)	0.4722(4)	0.25156(12)	0.0583(8)
C1	0.7707(5)	0.2525(5)	0.13838(18)	0.0450(11)
C2	0.8460(6)	0.1797(5)	0.1615(2)	0.0594(13)
H2	0.908074	0.226879	0.194304	0.071*
C3	0.8304(7)	0.0375(6)	0.1365(2)	0.0721(16)
H3	0.882248	−0.009509	0.152570	0.087*
C4	0.7395(7)	−0.0336(6)	0.0886(2)	0.0760(16)
H4	0.729023	−0.128992	0.072001	0.091*
C5	0.6639(7)	0.0356(6)	0.0652(2)	0.0760(17)
H5	0.601326	−0.013284	0.032631	0.091*
C6	0.6794(6)	0.1782(6)	0.0895(2)	0.0614(14)
H6	0.628091	0.224708	0.072767	0.074*
C7	0.9855(5)	0.6811(5)	0.1471(2)	0.0502(12)
C8	1.1274(6)	0.7718(6)	0.1766(2)	0.0712(15)
H8	1.142122	0.743147	0.210705	0.085*
C9	1.2495(7)	0.9051(7)	0.1572(3)	0.099(2)
H9	1.345737	0.964272	0.177731	0.119*
C10	1.2279(8)	0.9497(7)	0.1074(3)	0.096(2)
H10	1.309054	1.040207	0.094298	0.115*
C11	1.0883(8)	0.8619(7)	0.0772(2)	0.0799(17)
H11	1.073694	0.892270	0.043508	0.096*
C12	0.9684(6)	0.7283(6)	0.0964(2)	0.0623(13)
H12	0.873736	0.668050	0.075064	0.075*
C13	0.5864(5)	0.4788(5)	0.18301(18)	0.0488(11)
C14	0.5735(6)	0.6072(6)	0.1678(2)	0.0662(14)
H14	0.661490	0.691961	0.154741	0.079*
C15	0.4337(8)	0.6117(7)	0.1717(3)	0.0829(18)
H15	0.428302	0.699057	0.161258	0.100*
C16	0.3040(8)	0.4900(9)	0.1905(2)	0.0827(18)
H16	0.210270	0.494593	0.193370	0.099*
C17	0.3097(7)	0.3601(8)	0.2054(2)	0.0880(19)
H17	0.219977	0.275838	0.217922	0.106*
C18	0.4518(6)	0.3550(6)	0.2018(2)	0.0672(14)
H18	0.455792	0.266856	0.212119	0.081*
C19	0.8896(6)	0.8033(5)	0.31503(18)	0.0494(11)
C20	1.0294(7)	0.9475(6)	0.3240(2)	0.0724(15)
H20	1.123570	0.949060	0.333420	0.087*
C21	1.0307(8)	1.0894(6)	0.3190(3)	0.0844(18)
H21	1.124516	1.185807	0.326410	0.101*
C22	0.8952(8)	1.0876(7)	0.3032(2)	0.0797(17)
H22	0.896994	1.182755	0.298683	0.096*
C23	0.7574(8)	0.9484(7)	0.2941(2)	0.0840(18)
H23	0.664722	0.948369	0.283660	0.101*
C24	0.7534(6)	0.8050(6)	0.3003(2)	0.0663(14)
H24	0.657702	0.709820	0.294361	0.080*
C25	0.6856(5)	0.4172(5)	0.36092(18)	0.0430(10)
C26	0.6049(6)	0.2606(6)	0.3373(2)	0.0596(13)
H26	0.639086	0.231632	0.304773	0.071*
C27	0.4745(6)	0.1467(6)	0.3611(3)	0.0740(16)
H27	0.423177	0.041636	0.344971	0.089*
C28	0.4205(6)	0.1877(7)	0.4084(3)	0.0760(16)
H28	0.331648	0.111478	0.424213	0.091*
C29	0.4989(7)	0.3419(7)	0.4320(2)	0.0755(16)
H29	0.463067	0.370818	0.464156	0.091*
C30	0.6313(6)	0.4561(6)	0.4084(2)	0.0629(14)
H30	0.683825	0.560466	0.425242	0.075*
C31	1.1138(5)	0.6321(5)	0.35928(18)	0.0465(11)
C32	1.1920(6)	0.5598(6)	0.3327(2)	0.0652(14)
H32	1.146907	0.494090	0.299789	0.078*
C33	1.3337(7)	0.5829(7)	0.3536(3)	0.0838(18)
H33	1.384340	0.533117	0.335073	0.101*
C34	1.4016(6)	0.6796(7)	0.4021(3)	0.0793(17)
H34	1.497876	0.694887	0.416622	0.095*
C35	1.3280(6)	0.7523(6)	0.4286(2)	0.0726(16)
H35	1.375205	0.819296	0.461043	0.087*
C36	1.1835(6)	0.7285(5)	0.4081(2)	0.0570(13)
H36	1.133053	0.777486	0.427241	0.068*
C37	0.8145(8)	0.6751(8)	0.9459(2)	0.0862(19)
H37	0.881713	0.768406	0.930731	0.103*
C38	0.6741(6)	0.6526(6)	0.9661(2)	0.0630(14)
H38	0.644667	0.730394	0.964317	0.076*
C39	0.5759(5)	0.5146(6)	0.98902(18)	0.0500(12)
C40	0.8552(8)	0.5590(9)	0.9484(2)	0.092(2)
H40	0.950100	0.571390	0.934823	0.110*
C41	0.7534(8)	0.4252(8)	0.9710(3)	0.0854(18)
H41	0.781152	0.346075	0.972330	0.103*
C42	0.1908(8)	0.8771(9)	0.5585(2)	0.0840(18)
H42	0.289069	0.907104	0.575682	0.101*
C43	0.1631(6)	0.9876(7)	0.5369(2)	0.0667(14)
H43	0.243221	1.093436	0.538967	0.080*
C44	0.0179(5)	0.9424(5)	0.51227(17)	0.0487(11)
C45	0.0727(9)	0.7232(8)	0.5544(2)	0.0873(19)
H45	0.088482	0.645672	0.568524	0.105*
C46	−0.0678(8)	0.6853(7)	0.5296(3)	0.0856(18)
H46	−0.147923	0.579405	0.527268	0.103*

are much smaller than the reported in literature [14, 15]. The angle of Sn—O—Sn is 131.46(19)°. Interestingly, the angles of O—Sn—C and C—Sn—C are from 100° to 113°, which are closer to the regular tetrahedral configuration. Finally it should be mentioned that solvent-free parent structure is known for decades [16].

Acknowledgements: Financial support from the Shandong Provincial Higher Education Research and Development Program Project (No. J18KB049) and Talent Foundation of Taishan University (No. Y2015-1-002) are gratefully acknowledged.

References

1. Bruker. APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, WI, USA (2004).
2. Sheldrick, G. M.: Crystal structure refinement with SHELXL. Acta Crystallogr. **C71** (2015) 3–8.
3. Lee, M. R.; Chen, C. Y.: Organotin compounds analysis. Appl. Organomet. Chem. **11** (2012) 269–287.

4. Ma, C. L.; Zhang, J. H.; Jiang, Q.; Zhang, R. F.: Syntheses and crystal structures of three diorganotin(IV) macrocycles. Polyhedron **23** (2004) 2791–2797.
5. Ma, C. L.; Qin, J.; Zhang, R. F.: Syntheses, characterizations and crystal structures of new organotin complexes with 2-mercapto-6-nitrobenzothiazole. Polyhedron **23** (2004) 779–786.
6. Teoh, S. G.; Ang, S. H.; Looi, S. H.; Keok, C. A.; Teo, S. B.; Declercq, J. P.: Synthesis and crystal structure of di-n-butylbis(2-amino-5-chlorobenzoato)tin(IV). J. Organomet. Chem. **523** (1996) 75–78.
7. Sandhu, G. K.; Sharma, N.; Tiekink, E. R. T.: Structural chemistry of organotin carboxylates: VIII. synthesis and spectroscopic properties of diorganotin(IV) complexes with thiophenoxyacetic acid. X-ray crystal structures of $[nPr_2Sn(O_2CCH_2SPh)_2]$ and $\{[R_2Sn(O_2CCH_2SPh)]_2O\}_2$, R = nPr, nBu. J. Organomet. Chem. **403** (1991) 119–131.
8. Gielen, M.: Synthesis, characterization and *in-vitro* antitumour activity of dibutyltin carboxylates involving the perfluorophenyl moiety: crystal structure of the dimeric bis[(4-fluoro- and pentafluoro-benzoato)di-n-butyltin] oxides. Appl. Organomet. Chem. **9** (1995) 639–648.
9. Gielen, M.; Biesemans, M.; Khlou, A. E.; Meunier-Piret, J.; Kayser, F.; Willem, R.: Diorganotin difluorobenzoates: synthesis, spectroscopic characterization and *in vitro* antitumour activity. X-ray structure determination of bis[di-n-butyl(2,6-difluorobenzoato)tin] oxide. J. Fluorine Chem. **64** (1993) 279–291.
10. Wharf, I.; Lebuis, A. M.; Roper, G. A.: Studies in aryltin chemistry: part XIV. crystal and molecular structures of tris(o-methoxyphenyl)tin(IV) chloride, fluoride and oxide. Inorg. Chim. Acta **294** (1999) 224–231.
11. Lockhart, T. P.; Puff, H.; Schuh, W.; Reuter, H.; Mitchell, T. N.: Metal-metal spin coupling through chalcogenides. Correlation of $^2J(^{119}Sn, ^{119}Sn)$ with molecular structure. Crystal structure of $[(2\text{-}MeC_6H_4CH_2)_3Sn]_2O$ and $[(2\text{-}MeC_6H_4)_3Sn]_2O$. J. Organomet. Chem. **366** (1989) 61–72.
12. Pdělková, Z.; Weidlich, T.; Koláová, L.; Eisner, A.; Císaová, I.; Zevaco, T. A.; Rika, A.: Products of hydrolysis of *C,N*-chelated triorganotin(IV) chlorides and use of products as catalysts in transesterification reactions. J. Organomet. Chem. **692** (2007) 5633–5645.
13. Turek, J.; Padělková, Z.; Ernoek, Z.; Erbena, M.; Lyka, A.; Nechaev, M. S.; Císaová, I.; Rika, A.: *C,N*-chelated hexaorganodistannanes, and triorganotin(IV) hydrides and cyclopentadienides. J. Organomet. Chem. **694** (2009) 3000–3007.
14. Ma, C. L.; Sun, J. S.; Zhang, R. F.; Wang, D. Q.: Self-assembled diorganotin(IV) moieties with 2,3,4,5-tetrafuorobenzoic acid: syntheses, characterizations and crystal structures. Inorg. Chim. Acta **359** (2006) 4179–4190.
15. Ma, C. L.; Sun, J. S.; Zhang, R. F.: Trinuclear organooxotin assemblies from solvothermal synthesis reaction: crystal structure, hydrogen bonding and π–π stacking interaction. J. Mol. Struct. **833** (2007) 203–207.
16. Glidewell, C.; Liles, D. C.: Oxobis[triphenyltin(IV)]. Acta Crystallogr. **B34** (1978) 1693–1695.