

04) and thank Dr Yu Wang (National Taiwan University, Taipei, Taiwan) for her kind help in using the diffractometer and the *Structure Determination Package*.

References

CHATT, J. & WATSON, H. R. (1961). *J. Chem. Soc.* pp. 4980–4988.

Enraf-Nonius (1979). *Structure Determination Package*. Enraf-Nonius, Delft, The Netherlands.

FAVAS, M. C., KEPERT, D. L., SKELTON, B. W. & WHITE, A. H. (1980). *J. Chem. Soc. Dalton Trans.* pp. 447–453.

GABE, E. J. & LEE, F. L. (1981). *Acta Cryst.* A37, S339.

KING, R. B., KAPOOR, P. N. & KAPOOR, R. N. (1971). *Inorg. Chem.* 10, 1841–1850.

UENG, C.-H. & HWANG, G.-Y. (1991). *Acta Cryst.* C47, 522–525.

UENG, C.-H. & LEU, L.-C. (1991). *Acta Cryst.* C47, 725–728.

Acta Cryst. (1991). C47, 1593–1595

Structure of (2-Phenoxybenzoato)triphenyltin

BY SEIK WENG NG AND V. G. KUMAR DAS*

Institute of Advanced Studies and Department of Chemistry, University of Malaya, 59100 Kuala Lumpur, Malaysia

AND WAI-HING YIP AND THOMAS C. W. MAK*

Department of Chemistry, The Chinese University of Hong Kong, Shatin, New Territories, Hong Kong

(Received 7 November 1990; accepted 28 January 1991)

Abstract. [Sn(C₁₃H₉O₃)(C₆H₅)₃], $M_r = 563.24$, monoclinic, $P2_1/n$, $a = 15.447$ (3), $b = 10.172$ (1), $c = 17.271$ (4) Å, $\beta = 109.01$ (1)°, $V = 2565.6$ (7) Å³, $Z = 4$, $D_x = 1.46$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 10.25$ cm⁻¹, $F(000) = 1136$, $T = 298$ K, $R = 0.032$ for 3051 $|F_o| \geq 4\sigma|F_o|$ reflections. The four-coordinate Sn atom exists in a distorted tetrahedral environment.

Introduction. Both steric and electronic factors determine whether a triphenyltin benzoate will adopt a monomeric or a polymeric structure. Four-coordinate structures are generally adopted by the esters of those benzoic acids whose pK_a values exceed 3.0 (Molloy, Blunden & Hill, 1988). Steric factors dominate when the acid carries an *ortho* substituent, as for carboxylate-bridged (2-chlorobenzoato)triphenyltin (Holmes, Day, Chandrasekhar, Vollano & Holmes, 1986). (2-Methoxybenzoato)triphenyltin (Vollano, Day, Rau, Chandrasekhar & Holmes, 1984) is a four-coordinate molecule, and the present study of the 2-phenoxybenzoato analogue was carried out to ascertain whether replacing the methyl group by the more bulky phenyl group would lead to structural differences.

Experimental. Triphenyltin hydroxide (0.11 g, 0.3 mmol) and 2-phenoxybenzoic acid (0.11 g,

0.5 mmol) were dissolved in a small volume of 80% aqueous ethanol and the mixture refluxed for 1 h. Slow evaporation of the clear solution over a day afforded colorless prismatic crystals.

A crystal measuring 0.08 × 0.16 × 0.20 mm was mounted on a Nicolet R3m/V diffractometer for the diffraction study. Unit-cell dimensions were measured from 21 selected strong $5.05 \leq 2\theta \leq 17.33^\circ$ reflections and intensity data were collected at room temperature (298 K) using the ω -scan mode. The data set was processed with a profile-fitting procedure (Diamond, 1969) and corrected for absorption (Kopfmann & Huber, 1968) (transmission factors 0.848 to 0.920). The maximum 2θ angle was 50° , with hkl ranges h 0 to 19, k 0 to 13 and l -21 to 21; 4469 reflections were collected, of which 3051 had $|F_o| \geq 4\sigma|F_o|$. Two standard reflections (22 $\bar{2}$, $\bar{2}14$) monitored after every 125 data showed an intensity variation of $\pm 2\%$. The structure was solved by the heavy-atom method and refined (on F) by full-matrix least squares with anisotropic temperature factors for all non-H atoms. The H atoms of the phenyl groups were generated geometrically (C—H = 0.96 Å) and assigned an isotropic temperature factor of 0.08 Å². Computations were performed on a DEC MicroVAX II computer with the *SHELXTL-Plus* program package (Sheldrick, 1985). Analytical expressions of atomic scattering factors were used, and anomalous-dispersion corrections were incorporated (*International Tables for X-ray Crystallography*, 1974, Vol.

* Authors to whom correspondence should be addressed.

Table 1. Atomic coordinates ($\times 10^5$ for Sn; $\times 10^4$ for other atoms) and equivalent isotropic temperature factors ($\text{\AA}^2 \times 10^4$ for Sn; $\text{\AA}^2 \times 10^3$ for other atoms)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Sn(1)	27920 (2)	22362 (3)	4425 (2)	410 (1)
O(1)	992 (2)	2834 (3)	-579 (2)	62 (1)
O(2)	2168 (2)	4034 (3)	146 (2)	51 (1)
O(3)	2286 (2)	6137 (3)	-813 (2)	58 (1)
C(1)	1355 (3)	3905 (5)	-396 (3)	45 (2)
C(2)	894 (3)	5144 (5)	-782 (3)	47 (2)
C(3)	-60 (3)	5207 (5)	-1016 (3)	63 (2)
C(4)	-517 (4)	6342 (6)	-1344 (3)	74 (3)
C(5)	-43 (4)	7424 (6)	-1438 (3)	73 (3)
C(6)	897 (4)	7385 (5)	-1231 (3)	65 (2)
C(7)	1358 (3)	6242 (5)	-911 (3)	49 (2)
C(8)	2915 (3)	6788 (5)	-172 (3)	50 (2)
C(9)	2712 (3)	7212 (5)	501 (3)	59 (2)
C(10)	3393 (4)	7829 (5)	1128 (3)	74 (2)
C(11)	4247 (4)	7996 (6)	1082 (5)	92 (3)
C(12)	4446 (4)	7564 (6)	401 (5)	89 (3)
C(13)	3784 (4)	6943 (5)	-233 (4)	69 (3)
C(14)	4042 (3)	2910 (5)	1293 (2)	44 (2)
C(15)	4302 (3)	4218 (5)	1325 (3)	56 (2)
C(16)	5127 (3)	4639 (5)	1878 (3)	69 (2)
C(17)	5693 (3)	3762 (6)	2406 (3)	67 (2)
C(18)	5446 (3)	2477 (5)	2387 (3)	62 (2)
C(19)	4637 (3)	2038 (5)	1837 (3)	51 (2)
C(20)	2108 (3)	935 (4)	1004 (3)	46 (2)
C(21)	2375 (3)	836 (5)	1857 (3)	58 (2)
C(22)	1933 (4)	-58 (6)	2213 (3)	72 (3)
C(23)	1247 (4)	-844 (6)	1740 (4)	76 (3)
C(24)	976 (3)	-763 (5)	897 (4)	68 (3)
C(25)	1405 (3)	136 (5)	537 (3)	54 (2)
C(26)	2950 (3)	1617 (4)	-674 (2)	41 (2)
C(27)	3092 (3)	304 (5)	-798 (3)	54 (2)
C(28)	3249 (4)	-109 (5)	-1508 (3)	65 (2)
C(29)	3259 (3)	784 (6)	-2096 (3)	67 (2)
C(30)	3117 (3)	2095 (6)	-1981 (3)	63 (2)
C(31)	2968 (3)	2520 (4)	-1274 (3)	51 (2)

Table 2. Bond lengths (\AA) and bond angles ($^\circ$)

Sn(1)—O(2)	2.053 (3)	Sn(1)—C(14)	2.123 (4)
Sn(1)—C(20)	2.116 (5)	Sn(1)—C(26)	2.117 (5)
O(1)—C(1)	1.220 (6)	O(2)—C(1)	1.305 (5)
O(3)—C(7)	1.392 (6)	O(3)—C(8)	1.381 (5)
C(1)—C(2)	1.494 (6)	C(2)—C(3)	1.397 (7)
C(2)—C(7)	1.384 (7)	C(3)—C(4)	1.375 (8)
C(4)—C(5)	1.360 (9)	C(5)—C(6)	1.378 (9)
C(6)—C(7)	1.382 (7)	C(8)—C(9)	1.369 (8)
C(8)—C(13)	1.389 (8)	C(9)—C(10)	1.389 (7)
C(10)—C(11)	1.358 (10)	C(11)—C(12)	1.380 (12)
C(12)—C(13)	1.384 (8)	C(14)—C(15)	1.385 (7)
C(14)—C(19)	1.398 (6)	C(15)—C(16)	1.388 (6)
C(16)—C(17)	1.368 (7)	C(17)—C(18)	1.358 (8)
C(18)—C(19)	1.374 (6)	C(20)—C(21)	1.398 (6)
C(20)—C(25)	1.385 (6)	C(21)—C(22)	1.394 (9)
C(22)—C(23)	1.365 (8)	C(23)—C(24)	1.379 (9)
C(24)—C(25)	1.390 (8)	C(26)—C(27)	1.382 (7)
C(26)—C(31)	1.392 (6)	C(27)—C(28)	1.390 (8)
C(28)—C(29)	1.366 (8)	C(29)—C(30)	1.376 (9)
C(30)—C(31)	1.382 (7)		
O(2)—Sn(1)—C(14)	97.6 (1)	O(2)—Sn(1)—C(20)	114.0 (2)
O(2)—Sn(1)—C(26)	102.8 (1)	C(14)—Sn(1)—C(20)	111.3 (2)
C(14)—Sn(1)—C(26)	112.2 (2)	C(20)—Sn(1)—C(26)	117.0 (2)
Sn(1)—O(2)—C(1)	110.7 (3)	C(7)—O(3)—C(8)	119.1 (4)
O(1)—C(1)—O(2)	122.0 (4)	O(1)—C(1)—C(2)	121.7 (4)
O(1)—C(1)—C(2)	116.3 (4)	C(1)—C(2)—C(3)	118.2 (4)
C(1)—C(2)—C(7)	123.8 (4)	C(3)—C(2)—C(7)	118.0 (4)
C(2)—C(3)—C(4)	120.6 (5)	C(3)—C(4)—C(5)	120.3 (5)
C(4)—C(5)—C(6)	120.5 (5)	C(5)—C(6)—C(7)	119.3 (5)
O(3)—C(7)—C(2)	119.0 (4)	O(3)—C(7)—C(6)	119.3 (5)
C(2)—C(7)—C(6)	121.1 (5)	O(3)—C(8)—C(9)	122.5 (5)
O(3)—C(8)—C(13)	115.9 (5)	C(9)—C(8)—C(13)	121.5 (4)
C(8)—C(9)—C(10)	118.7 (5)	C(9)—C(10)—C(11)	120.9 (6)
C(10)—C(11)—C(12)	120.1 (6)	C(11)—C(12)—C(13)	120.4 (6)
C(8)—C(13)—C(12)	118.4 (6)	Sn(1)—C(14)—C(15)	121.6 (3)
Sn(1)—C(14)—C(19)	120.7 (3)	C(15)—C(14)—C(19)	117.6 (4)
C(14)—C(15)—C(16)	120.9 (4)	C(15)—C(16)—C(17)	120.0 (5)
C(16)—C(17)—C(18)	119.9 (4)	C(17)—C(18)—C(19)	121.0 (4)
C(14)—C(19)—C(18)	120.6 (4)	Sn(1)—C(20)—C(21)	120.5 (3)
Sn(1)—C(20)—C(25)	120.9 (4)	C(21)—C(20)—C(25)	118.6 (5)
C(20)—C(21)—C(22)	119.5 (4)	C(21)—C(22)—C(23)	120.9 (5)
C(22)—C(23)—C(24)	120.4 (6)	C(23)—C(24)—C(25)	119.1 (5)
C(20)—C(25)—C(24)	121.5 (5)	Sn(1)—C(26)—C(27)	120.0 (3)
Sn(1)—C(26)—C(31)	121.2 (3)	C(27)—C(26)—C(31)	118.7 (4)
C(26)—C(27)—C(28)	120.7 (5)	C(27)—C(28)—C(29)	120.2 (5)
C(28)—C(29)—C(30)	119.7 (5)	C(29)—C(30)—C(31)	120.7 (5)
C(26)—C(31)—C(30)	120.0 (4)		

IV, pp. 55, 99, 149). The final least-squares cycle was calculated with 316 variables. The residuals are $R = 0.032$, $wR = 0.035$ $\{w = [\sigma(F_o) + 0.0004|F_o|]^2\}^{-1}$, $S = 0.9777$ and $(\Delta/\sigma)_{max} = 0.001$; residual extrema in the final difference map ranged from $+0.37$ to -0.30 e \AA^{-3} . Fractional coordinates are given in Table 1* and bond distances and angles in Table 2.

Discussion. (2-Phenoxybenzoato)triphenyltin crystallizes as a four-coordinate molecule (Fig. 1) whose geometry at Sn is a tetrahedron that is distorted owing to an intramolecular acyl O...Sn contact of $2.832(5) \text{ \AA}$. The dihedral angle between the CO_2 plane and the plane defined by the C(2)—C(7) atoms is $31.5(6)^\circ$; this corresponds to a rotation about the C(1)—C(2) bond, in the absence of which crowding between O(2) and O(3) would be severe. The two phenyl rings of the anionic group are not conjugated as they are tilted by $84.6(6)^\circ$. The bond distances and angles are similar to those found in (2-methoxybenzoato)triphenyltin (Vollano *et al.*, 1984).

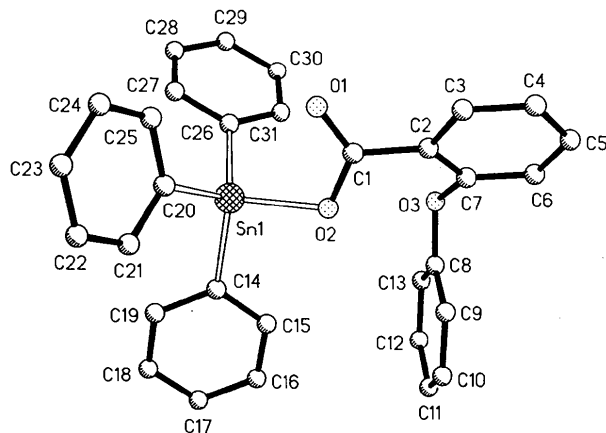


Fig. 1. Molecular diagram with labeling scheme for (2-phenoxybenzoato)triphenyltin.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53955 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

We thank the University of Malaya (Vote F PJP286/89), the National Science Council for R&D, Malaysia (grant No. 2-07-04-06) and the UPGC

Grant for R&D, Hong Kong (acc. No. 221300010) for supporting this work.

References

DIAMOND, R. (1969). *Acta Cryst.* **A25**, 43–55.

HOLMES, R. R., DAY, R. O., CHANDRASEKHAR, V., VOLLANO, J. F. & HOLMES, J. M. (1986). *Inorg. Chem.* **25**, 2490–2494.

KOPFMANN, G. & HUBER, R. (1968). *Acta Cryst.* **A24**, 348–351.

MOLLOY, K. C., BLUNDEN, S. J. & HILL, R. (1988). *J. Chem. Soc. Dalton Trans.* pp. 1259–1266.

SHELDRIK, G. M. (1985). In *Crystallographic Computing 3: Data Collection, Structure Determination, Proteins, and Databases*, edited by G. M. SHELDRIK, C. KRÜGER & R. GODDARD, pp. 175–189. Oxford Univ. Press.

VOLLANO, J. F., DAY, R. O., RAU, D. N., CHANDRASEKHAR, V. & HOLMES, R. R. (1984). *Inorg. Chem.* **23**, 3153–3160.

Acta Cryst. (1991). **C47**, 1595–1597

Structure of Aquabis(sulfato)tetrakis(urea)thorium(IV) Dihydrate

BY J. HABASH* AND R. L. BEDDOES

Department of Chemistry, University of Manchester, Manchester M13 9PL, England

AND A. J. SMITH

Department of Chemistry, The University, Sheffield S3 7HF, England

(Received 22 November 1990; accepted 29 January 1991)

Abstract. $[\text{Th}(\text{SO}_4)_2(\text{CH}_4\text{N}_2\text{O})_4(\text{H}_2\text{O})] \cdot 2\text{H}_2\text{O}$, $M_r = 718.42$, monoclinic, $C2/c$, $a = 16.17$ (1), $b = 8.15$ (1), $c = 32.01$ (1) Å, $\beta = 93.52$ (4)°, $V = 4210$ (6) Å³, $Z = 8$, $D_m = 2.255$, $D_x = 2.267$ g cm⁻³, Mo $K\alpha$ radiation (graphite monochromator, $\lambda = 0.71069$ Å), $\mu = 76$ cm⁻¹, $F(000) = 2752$, room temperature, final $R = 0.023$ for 4011 unique observed X-ray reflections. The thorium atom is bonded to nine oxygen atoms: four of these are contributed by two bidentate sulfato ligands and a further four by urea ligands. A water oxygen atom completes the tricapped trigonal prismatic (3/3/3) thorium coordination polyhedron (idealized symmetry D_{3h}).

Introduction. Thorium has extensive coordinating power and chiefly exhibits nine- or ten-coordination (Habash & Smith, 1983, 1990). A series of sulfato-aqua compounds of thorium were prepared and studied by Evstaféva, Molodkin, Dvoryantseva, Ivanova & Struchkova (1966). The infrared absorption spectra of urea-containing nitrato and halogeno complexes of thorium were interpreted by Petrov, Molodkin, Ivanova & Saralidze (1969) who concluded that the thorium is bonded to urea through oxygen atoms. The aim of the present work was to establish whether urea-sulfato complexes of thorium contain O- or N-bonded urea.

Experimental. Colourless prismatic crystals, elongated along [010], were obtained by slow evaporation

of a solution containing $\text{Th}(\text{SO}_4)_2 \cdot 4\text{H}_2\text{O}$ and urea, $\text{CO}(\text{NH}_2)_2$, in a molar ratio of 1:8, at room temperature. Chemical analysis confirmed the formula {found: Th 32.10, S 8.27, N 15.98, C 6.74, H 3.07%; calculated for $[\text{Th}(\text{SO}_4)_2 \cdot [\text{CO}(\text{NH}_2)_2]_4 \cdot \text{H}_2\text{O}] \cdot 2\text{H}_2\text{O}$: Th 32.29, S 8.92, N 15.59, C 6.69, H 3.08%}. Thorium was determined gravimetrically by precipitation as oxalate and ignition to the dioxide. Sulfur, nitrogen, carbon and hydrogen were estimated by standard microanalytical techniques. X-ray data from a small (approximately $0.11 \times 0.08 \times 0.29$ mm) crystal, mounted parallel to [010], were collected on a Rigaku AFC6S diffractometer operating in the ω -scan mode using monochromated Mo $K\alpha$ radiation. The unit-cell parameters were determined by least squares from ω measurements of 25 carefully centred reflections in the range $9.56 < 2\theta < 19.22^\circ$. Two octants of reciprocal space, hkl and $hk\bar{l}$ (h 0 to 19, k 0 to 9 and \bar{l} -38 to 37), were explored up to $2\theta = 50^\circ$. 4169 measurements were made on 4011 reflections. The density was measured by flotation in a mixture of iodobenzene and diiodomethane. After corrections, intensities of 3085 independent reflections greater than $3\sigma(I)$ above background were obtained for structure determination. Three standard reflections were monitored every 150 measured reflections and showed no sign of decay. Corrections for Lorentz-polarization effects, secondary extinction and absorption were carried out (transmission range 76 to 100%). Neutral atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol.

* To whom correspondence should be addressed.