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Crystallographic report

Bis[3-(tri-*p*-tolyl)germyl-3-(*o*-tolyl)-propionato]dibutyltin(IV)

Imtiaz-ud-Din[1], M. Mazhar[1]*, Sarim Dastgir[1], Mary F. Mahon[2] and Kieran C. Molloy[2]

[1]Department of Chemistry, Quaid-i-Azam University, Islamabad 45320, Pakistan
[2]Department of Chemistry, University of Bath, Bath BA2 7AY, UK

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The crystal structure of $[(p\text{-}CH_3C_6H_4)_3GeCH(o\text{-}CH_3C_6H_4)CH_2CO_2]_2Sn(C_4H_9)_2$ consists of a monomer with the atoms of tin and germanium both occupying tetrahedral geometries. However, the tin atom is distorted towards a skew trapezoidal bipyramid geometry as a result of weakly chelating carboxylate ligands. Copyright © 2003 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; diorganotindicarboxylates; germanium

COMMENT

Diorganotin compounds containing germanium as a part of the carboxylate ligand have been synthesized in a continuation of our previous work.[1,2] The structure of bis[3-(tri-*p*-tolyl)germyl-3-(*o*-tolyl)-propionato]dibutyltin(IV) has been determined. The structure consists of a monomer with the germanium occupying a tetrahedral geometry (Fig. 1). The average bond angle around the germanium atom is 108.5°. The tin atom is chelated by the two asymmetrically coordinating carboxylate ligands and two butyl groups with an average bond angle around tin of 102.0°, which depicted distorted tetrahedral geometry. There is an indication of weak interactions of tin with O(2) [2.541(2) Å] and O(4) [2.694(3) Å], as manifested by the opening of the C(63)–Sn(1)–C(67) angle to 138.41(14)°. The tin atom geometry is thus best described as based on a skew trapezoidal bipyramid geometry.[3] The bond lengths of Sn(1)–O(1) and Sn(1)–O(3) are identical [2.106(2) Å] and the Sn(1)–C(63) [2.121(4) Å] and Sn(1)–C(67) [2.127(3) Å] bonds are normal. The weakly hexa-coordinated tin appears to be present in solution as indicated by the upfield ^{119}Sn NMR resonance at −147.2 ppm ($CDCl_3$).

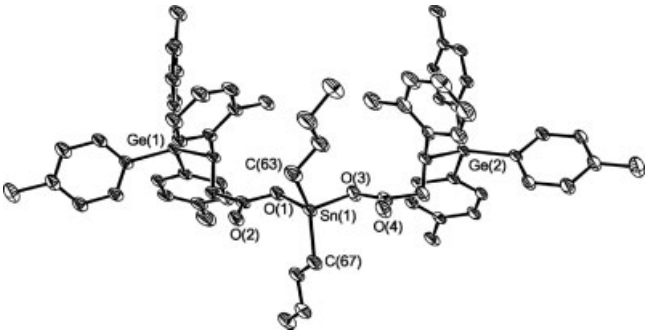

Figure 1. Molecular structure of bis[3-(tri-*p*-tolyl) germyl-3-(o-tolyl)-propionato]dibutyltin(IV). Selected geometric parameters: Ge(1)–C(11) 1.943(3) Å; O(1)–Sn(1)–O(3) 80.29(9), O(1)–Sn(1)–C(63) 100.63(12), O(1)–Sn(1)–C(67) 107.04(12), O(3)–Sn(1)–C(63) 112.84(12), O(3)–Sn(1)–C(67) 102.10(12)°.

EXPERIMENTAL

Stoichiometric amounts of [3-(tri-*p*-tolyl)germyl-3-*o*-tolylpropanoic acid (1.02 g, 2.0 mmol) and dibutyltin oxide (0.25 g, 1.0 mmol) were suspended in toluene (50 cm^3) and refluxed for 8 h. Water formed during the reaction was removed by a Dean and Stark apparatus; toluene was subsequently removed under vacuum and the crude product was recrystallized from chloroform/petroleum ether (3:1) to yield colourless crystals. M.p. 208–209 °C. IR (KBr, cm^{-1}) $\nu(COO)_{asy}$ 1625, $\nu(COO)_{sym}$ 1376, ν(Sn–O) 489, ν(Sn–C) 589, ν(Ge–C) 670. Crystallographic details: intensity data were collected at 150 K on a Nonius Kappa CCD diffractometer for a crystal 0.10 × 0.10 × 0.15 mm^3. $C_{70}H_{80}Ge_2O_4Sn$, $M = 1249.18$, triclinic, $P\bar{1}$, $a = 13.5560(3)$, $b = 13.7670(3)$, $c = 18.9780(4)$ Å, $\alpha = 97.456(1)$, $\beta =$

*Correspondence to: M. Mazhar, Department of Chemistry, Quaid-i-Azam University, Islamabad 45320, Pakistan.
E-mail: mazhar42pk@yahoo.com

94.669(1), $\gamma = 114.948(1)^\circ$, $V = 3147.4(1)$ Å^3, $Z = 2$, $\theta_{max} = 27.5^\circ$, 14 342 independent reflections, $R_1 = 0.068$ (all data), $wR_2 = 0.103$ (all data). Programs used: SHELXS86, SHELXS97, ORTEX95. CCDC deposition number 200 434.

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