Crystal Structure of Tricyclohexyltin 2,2,3,3-Tetramethylcyclopropanecarboxylate

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Figure 1: Molecular structure of $[\text{C}_3\text{H}_5\text{SnO}_2\text{CC}_3\text{H}(\text{CH}_3)_4]$. Key geometric parameters: Sn-O1 2.111 (2), Sn-C1 2.159(4), Sn-C7 2.164(3), Sn-C13 2.148(3), C19-O1 1.305(4), C19-O2 1.226(4) Å; C1-Sn-C7 123.02(14), C1-Sn-C13 112.67(15), C7-Sn-C13 116.01(13), O1-Sn-C1 101.80(13), O1-Sn-C7 103.82(12), O1-Sn-C13 92.47(11), H-atoms omitted for clarity.

COMMENT

Triorganotin /1-3/ and pyrethroids /4/ have been shown to be effective insecticides against mosquito larvae as well as adult mosquitoes. In an attempt to synthesize a more effective insecticide, a precursor of the acid moiety of pyrethroid, 2,2,3,3-tetramethyl cyclopropane carboxylic acid, was incorporated into a tricyclohexyltin molecule. It is hoped that the “designed” molecule will exhibit synergistic effects in its activity. The crystal structure analysis for tricyclohexyltin 2,2,3,3-tetramethylcyclopropanecarboxylate indicated that the molecule is monomeric with a distorted tetrahedral geometry as shown in Fig. 1. The tin

atom is coordinated to the carboxylate group *via* one of the oxygen atoms forming a Sn-O(1) bond of 2.111 (2) Å. The other Sn-O bond distances are over 3 Å indicating that no other significant interactions exist between these two atoms. However, the second O atom of the carboxylate group may have a steric influence and contribute to the distorted tetrahedral geometry of the molecule. The observed Sn-O value is similar to those found for other tricyclohexyltin carboxylates such as tricyclohexyltin 2-(*p*-chlorophenyl)-3-methylbutyrate /5/, 4-hydroxybenzoate /6/ and 2-[(*E*)-2-(2-hydroxy-5-methylphenyl)-1-diazenyl] benzoate /7/.

EXPERIMENTAL

Tricyclohexyltin hydroxide (5 mmol) was added, with stirring, to 50 mL of toluene. To this solution was added 2,2,3,3-tetramethylcyclopropanecarboxylic acid (5 mmol). The mixture was then refluxed for 1 h. A Dean-Stark trap was used to remove the water formed. The toluene was then removed under vacuum and a clear oil was obtained. A white solid formed upon refrigeration overnight. The solid was then dissolved in 95% ethanol, and upon slow evaporation, crystals suitable for X-ray diffraction analyses were obtained, mp 72-74 °C. The crystallographic analysis was done at the X-ray Structural Characterization Laboratory in the Chemistry Department at the University of Massachusetts, Amherst.

Table 1
Crystal data for Tricyclohexyltin 2,2,3,3-tetramethylcyclopropanecarboxylate

Formula	C ₂₆ H ₄₆ O ₂ Sn	Formula weight	509.32
Crystal system	Monoclinic	Crystal size, mm	0.50×0.40×0.40
Space group	<i>P</i> 2 ₁ / <i>c</i>	<i>a</i> , Å	10.3984(1)
<i>b</i> , Å	11.5462(1)	<i>c</i> , Å	22.5738(3)
β, °	99.7754(5)	<i>V</i>	2670.90(5)
<i>Z</i>	4	Diffractometer	Nonius Kappa CCD
Temperature	293(2)	μ (Mo-Kα), cm ⁻¹	9.74
<i>D</i> _{calc} , g cm ⁻³	1.267	<i>F</i> (000)	1072
θ _{max} , °	27.53	Reflns unique	6067
Reflns with <i>I</i> ≥ 2σ(<i>I</i>)	5046	No. parameter	252
Weighting scheme	1/[σ ² (<i>F</i> _o ²)+(0.0457 <i>P</i>) ² +3.1616 <i>P</i>] <i>P</i> =(<i>F</i> _o ² +2 <i>F</i> _c ²)/3	Goodness of fit	1.027
<i>R</i> (all data)	<i>R</i> 1 = 0.0497, <i>wR</i> 2 = 0.1066	ρ, e Å ⁻³	0.781
		Final <i>R</i> [<i>I</i> >2σ (<i>I</i>)]	<i>R</i> 1 = 0.0399, <i>wR</i> 2 = 0.1002
Programs used	SHELXS-86 /8/, SHELXL-97 /9/ and ORTEP /10/		
Deposition number	CCDC 246577		

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REFERENCES

1. G. Eng, C. Whitmyer, B. Sina and N. Ogwuru, *Main Group Met. Chem.*, **22**, 311 (1999).
2. N. Ogwuru, Q. Duong, X. Song and G. Eng, *Main Group Met. Chem.*, **24**, 775, (2001).
3. G. Eng, X. Song, Q. Duong, D. Strickman, J. Glass and L. May, *Appl. Organometal. Chem.*, **18**, 363 (2004).
4. R. J. Cremllyn, *Agrochemicals Preparation and Mode of Action*, John Wiley & Sons, Chichester, West Sussex, England, 1991; p. 53 and references therein.
5. X. Song, C. Cahill and G. Eng, *Appl. Organomet. Chem.*, **17**, 743 (2003).
6. X. Song, C. Cahill and G. Eng, *Main Group Met. Chem.*, **25**, 703 (2002).
7. R. Willem, I. Verbruggen, M. Gielen, M. Biesemans, B. Mahieu, T. S. B. Baul and E. R. T. Tiekink, *Organometallics*, **17**, 5758 (1998).
8. G. M. Sheldrick, *Acta Crystallogr.*, **A46**, 467 (1990).
9. G. M. Sheldrick, *SHELXL-97: Program for Crystal Structure Refinement*, University of Gottingen, 1997.
10. C. K. Johnson, ORTEP, Report ORNL-5138, Oak Ridge National Laboratory, TN, USA, 1976.