A RARE EXAMPLE OF AN UNSYMMETRICALLY SUBSTITUTED TETRAORGANODISTANNOXANE LADDER: $[\text{cyclo-Hex}_2(\text{OH})\text{SnOSn}(\text{Cl})\text{t-Bu}_2]_2$

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Figure 1. Molecular structure (25 % probability ellipsoids) of $[\text{cyclo-Hex}_2(\text{OH})\text{SnOSn}(\text{Cl})\text{t-Bu}_2]_2$. Symmetry transformations used to generate equivalent atoms: $a = 0.5 - X, 1.5 - Y, 1 - Z$; $b = -X, 1 - Y, -Z$. Selected bond distances (Å) and angles (°): Sn(1)-O(1) 2.169(3), Sn(1)-O(2) 2.035(3), Sn(1)-Cl(1) 2.562(1), Sn(1)-C(1) 2.161(5), Sn(1)-C(11) 2.176(6), Sn(2)-O(1) 2.191(3), Sn(2)-O(2) 2.063(3), Sn(2)-O(2a) 2.144(3), Sn(2)-Cl(1a) 3.692(1), Sn(2)-C(21) 2.128(5), Sn(2)-C(31) 2.142(6), Sn(3)-O(3) 2.052(3), Sn(3)-O(4) 2.171(3), Sn(3)-C(41) 2.198(6), Sn(3)-C(51) 2.207(6), Sn(3)-Cl(2) 2.551(1), Sn(4)-O(3) 2.055(3), Sn(4)-O(3b) 2.175(3), Sn(4)-O(4) 2.187(3), Sn(4)-C(61) 2.128(6), Sn(4)-C(71) 2.138(5), Sn(4)-Cl(2a) 3.840(1), O(1)-Sn(1)-Cl(1) 158.19(9), O(1)-Sn(1)-C(1) 95.28(17), O(1)-Sn(1)-C(11) 96.87(16), O(1)-Sn(1)-O(2) 73.49(11), C(1)-Sn(1)-C(11) 122.9(2), C(1)-Sn(1)-O(2) 121.5(2), C(11)-Sn(1)-O(2) 115.45(18), C(1)-Sn(1)-Cl(1) 93.85(16), C(11)-Sn(1)-Cl(1) 94.68(14), O(2)-Sn(1)-Cl(1) 84.85(8), O(1)-Sn(2)-O(2) 72.48(11), O(1)-Sn(2)-O(2a) 145.79(10), O(1)-Sn(2)-C(21) 95.05(17), O(1)-Sn(2)-C(31) 91.40(19), O(2)-Sn(2)-O(2a) 73.33(13), O(2)-Sn(2)-C(21) 116.28(17), O(2)-Sn(2)-C(31) 112.6(2), C(21)-Sn(2)-C(31) 130.4(2), C(21)-Sn(2)-O(2a) 98.76(17), C(31)-Sn(2)-O(2a) 102.84(17), O(3)-Sn(3)-O(4) 72.95(11), O(3)-Sn(3)-C(41) 114.88(18), O(3)-Sn(3)-C(51) 125.9(2), O(3)-Sn(3)-Cl(2) 86.12(8), O(4)-Sn(3)-C(41) 94.41(16), O(4)-Sn(3)-C(51) 97.85(16), O(4)-Sn(3)-Cl(2) 158.72(9), C(41)-Sn(3)-C(51) 119.0(2), C(41)-Sn(3)-Cl(2) 97.96(14), C(51)-Sn(3)-Cl(2) 91.23(14), O(3)-Sn(4)-O(4) 72.54(11), O(3)-Sn(4)-O(3b) 75.20(12), O(3b)-Sn(4)-O(4) 147.64(10), O(3)-Sn(4)-C(61) 113.4(2), O(3)-Sn(4)-C(71) 119.99(17), C(61)-Sn(4)-C(71) 126.6(2), C(61)-Sn(4)-O(3b) 97.83(18), C(71)-Sn(4)-O(3b) 96.67(18), C(61)-Sn(4)-O(4) 97.04(18), C(71)-Sn(4)-O(4) 97.23(18), Sn(1)-O(1)-Sn(2) 102.07(12), Sn(1)-O(2)-Sn(2) 111.66(13), Sn(1)-O(2)-Sn(2a) 141.55(14), Sn(2)-O(2)-Sn(2a) 106.67(13), Sn(3)-O(3)-Sn(4) 111.83(13), Sn(3)-O(3)-Sn(4b) 143.36(14), Sn(4)-O(3)-Sn(4b) 104.80(12), Sn(3)-O(4)-Sn(4) 102.63(12).

COMMENT

The title compound 1,1-dicyclohexyl-3,3-di-*t*-butyl-1-hydroxy-3-chlorodistannoxane was prepared by reaction of *cyclo*-(*t*-Bu₂SnO)₃ with (*cyclo*-Hex)₂SnCl₂ in dichloromethane in the presence of air moisture and single crystals were obtained from a toluene solution (m.p. > 250°C, $\nu(\text{OH})$ 3500 cm⁻¹, Elemental Analysis calcd for C₂₀H₄₁ClO₂Sn₂: C, 40.96; H, 7.05; Cl, 6.05. Found C, 41.10; H, 6.89; Cl, 6.22%). The unit cell contains two crystallographically independent dimers which are both centrosymmetric. As in related tetraorganodistannoxanes [1-3], the tin atoms Sn(2), Sn(2a) and Sn(4), Sn(4b), respectively, are incorporated in central Sn₂O₂ four-membered rings and the tin atoms Sn(1), Sn(1a) and Sn(3), Sn(3b), respectively, are located exocyclic to these rings. The Sn₄O₂Cl₄ structural motifs are planar to a deviation of fitted atoms of 0.034 Å and 0.065 Å, respectively. Both the exocyclic tin atoms Sn(1) and Sn(3) and the endocyclic tin atoms Sn(2) and Sn(4) each exhibit a distorted trigonal bipyramidal configuration (geometrical goodness [4] $\Delta\Sigma(\theta) = 86.5^\circ$ (Sn1), 84.5° (Sn3), 84.4° (Sn2), 90.3° (Sn4)) with the axial positions being occupied by Cl(1), O(1) and Cl(2), O(4) and O(1), O(2a) and O(4), O(3a), respectively. The equatorial positions are occupied by C(1), C(11), O(2) for Sn(1), C(41), C(51), O(3) for Sn(3), C(21), C(31), O(2) for Sn(2) and C(51), C(61), O(3) for Sn(4). Furthermore, there are intramolecular Sn(2)⋯Cl(1a) and Sn(4)⋯Cl(2a) distances of 3.692(1) and 3.840(1) Å, respectively, being shorter than the sum of the van der Waals radii [5] of tin (2.20 Å) and chlorine (1.7 – 1.9 Å) and apparently being related to the widening of the C(21)-Sn(2)-C(31) and C(61)-Sn(4)-C(71) angles to 130.4(2) and 126.6(2)°, respectively.

Table 1. Crystal data for $[\text{cyclo-Hex}_2(\text{OH})\text{SnOSn}(\text{Cl})t\text{-Bu}_2]_2$

Formula	C ₄₀ H ₈₂ Cl ₂ O ₄ Sn ₄	Formula weight	1170.70
Cryst syst	monoclinic	Crystal size, mm	0.20 x 0.10 x 0.10
Space group	C2/c	a, Å	40.685(1)
b, Å	11.935(1)	c, Å	28.476(1)
β, °	134.428(1)	V, Å ³	9874.3(9)
Temperature, K	173(1)	Z	8
μ (Mo-Kα)	2.142 mm ⁻¹	Diffractometer	Nonius KappaCCD
D _{calcd} , g cm ⁻³	1.575	F(000)	4688
θ _{max} , °	27.9	Reflns meas.	61645
Reflns unique, R _{int}	11711, 0.052	Reflns with I > 2σ (I)	4070
Weighting scheme	1/[σ ² (F _o ²)]	R(F) (I > 2σ (I))	0.032
Programs used	SHELXS97[6] SHELXL97[7] SHELXTL[8]	R _w (F ⁺) (all data)	0.070
		ρ, e Å ⁻³	0.91 and -0.58
		Deposition number	CCDC 183143

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