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Dinuclear Tin(II) Complex of a Bulky *cis*-Oxamide: Synthesis, Characterization, Crystal Structure, and DFT Studies

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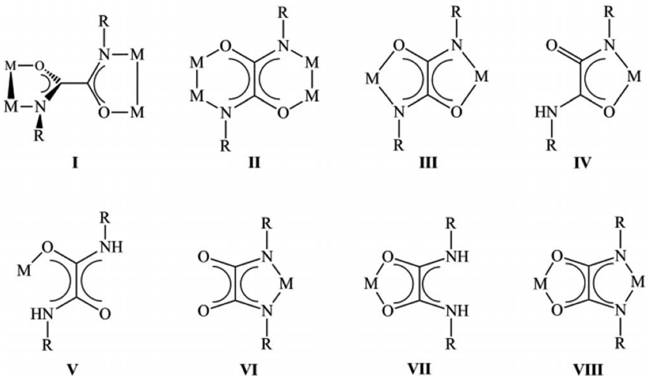
Dedicated to Professor Herbert W. Roesky on the Occasion of His Birthday

Keywords: Oxamides; Tin; ^{119}Sn NMR spectroscopy; Crystal structure; Density functional calculations

Abstract. The reaction of the di-lithiated oxamide of **1** with two equivalents of $SnCl_2$ provided the tin *trans*-oxamide **3**. In solution, spectroscopic analysis suggests exclusively the formation of a *trans*-oxamide (*trans*-**3**). However, the solid state shows an atypical *cis*-oxamide (*cis*-**3**), where the oxamide fragment acts as an *anti-Janus head* ligand. An ^{119}Sn-NMR variable temperature experiment ([D$_8$]THF) of the *trans*-oxamide (*trans*-**3**) was performed however, at lower temperature no additional signal was observed, which confirmed the absence of a dynamic equilibrium. Dispersion-corrected density functional calculations revealed that the *cis* conformation of this tin(II) oxamide complex is more stable than the *trans* isomer by 1.4 kcal·mol^{-1}.

Introduction

N,*N*′-Disubstituted oxamides have been of great importance to crystal engineering,[1] pharmaceutical,[2] and coordination chemistry.[3] Oxamides as ligands show a diversity of bonding modes (Scheme 1)[4–6] and several synthetic advantages as: (*i*) high yields, (*ii*) simple synthetic routes, (*iii*) short time reactions, and (*iv*) easy isolation. Particularly, *trans*-oxamide (**III**) and *cis*-oxamide (**VIII**) could be considered as *Janus head*[7] and *anti-Janus head* complexes, respectively. Both conformations with transition metal oxamides have been reported.[8] Oxamides with *anti-Janus head* conformation are typically synthesized in a step-wise pathway.[9] On the other hand, complexes with main group elements (B, Al, Ga, Si, Ge, Sn, and Sb) in *trans* conformation were synthesized,[10–15] whereas those with *cis* conformation are quite scarce. In 1977 *Fischer* and *Stezowski* reported that both isomers **III** and **VIII** come from the same reaction and the configurational isomerism depends upon both size and nature of the substituents at the oxamide nitrogen and the gallium atom.[16] Based on that, *Ziemkowska* et al. reinvestigated the reactions of aluminum oxamide complexes varying the substituents on nitrogen atoms and a mixture of two configurational isomers was determined by ^{13}C-NMR spectroscopy.[17]



Scheme 1. Different coordination modes for oxamide ligands.

Thus, in this contribution we describe the synthesis and molecular structure of the first dinuclear Sn^{II}-*cis*-oxamide complex. ^{119}Sn VT-NMR and DFT calculations show that dispersion in such systems is fundamental to describe correctly the energetic of the title system.

Results and Discussion

Reaction of oxalylchloride with the corresponding disubstituted aniline provided the sterically crowded oxamides **1** and **2** in good yields (Scheme 2) as colorless sharp melting crystalline materials with good solubility in THF, $CHCl_3$, and

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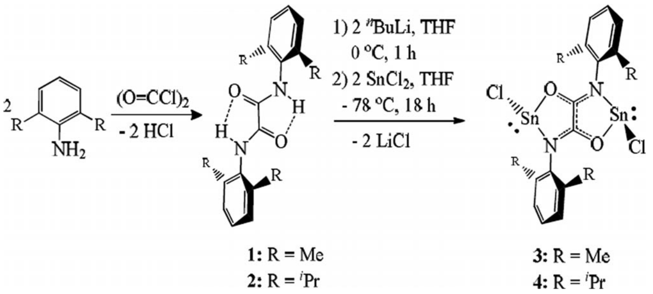
CH_2Cl_2. The molecular structure of **1**, as determined by single-crystal X-ray diffraction analysis, is depicted in Figure 1, (selected bond lengths and angles are given in the Supporting Information). The crystal structure of the oxamide **1**, crystallized from slow evaporation at room temperature after one week, shows *trans* conformation with C_2 symmetry. The low symmetry is due to a non-planar array of the aromatic rings (torsion angle C1–N2–C3–C8 = 66.7°). The C1–O1 [1.208(4) Å], C1–N2 [1.327(4) Å], and C1–C11 [1.509(4) Å] bond lengths are slightly shorter than those of similar already reported oxamides $(Me_3C_6H_2NHCO)_2$, [C–O 1.2188(4) Å, C–N 1.337(5) Å, C–C 1.5338(3) Å].[18] Reaction of the di-lithiated oxamides of **1** and **2** with two equivalents of $SnCl_2$ in THF at low temperature provided, after work-up, compounds *trans*-**3** and *trans*-**4**, respectively, as amorphous materials in approximately 52 % yield (Scheme 2).



Scheme 2. Synthesis of compounds **1**–**4**.

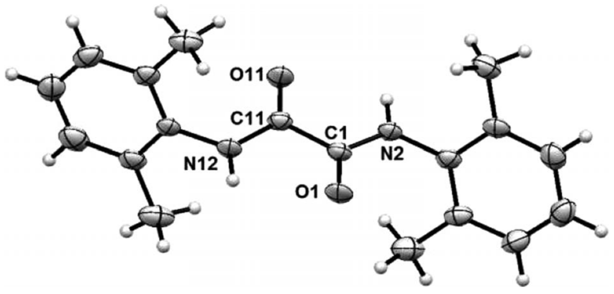
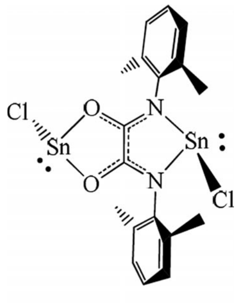


Figure 1. ORTEP view of **1**. Thermal ellipsoids are drawn at 30 % probability level.

The room temperature ^{119}Sn NMR spectra of *trans*-**3** and *trans*-**4**, in C_6D_6, exhibit only one sharp resonance at δ = –147 ppm ($\Delta\nu_{1/2}$ = 79 Hz) and δ = –190 ppm ($\Delta\nu_{1/2}$ = 84 Hz), respectively, which indicates a tetracoordinate tin atom with *pseudo*-square pyramidal arrangement.[19] At – 80 °C, in $[D_8]$THF the signals are shifted to δ = –162 ppm ($\Delta\nu_{1/2}$ = 596 Hz) and δ = –226 ppm ($\Delta\nu_{1/2}$ = 432 Hz), respectively. The observation of exclusively one resonance for each solution is consistent with the existence in solution of a single species, either *trans*-**3** or *trans*-**4**. Even though *cis*-**3** (Scheme 3) was never detected in solution, this complex crystallized from the THF solution of *trans*-**3** at – 20 °C after 2 weeks. The formation of *cis*-**3** might be explained on the basis of a *trans*-**3** isomerization by simultaneous Sn–O and Sn–N bond rupture, rotation about the C–C bond and reformation of Sn–O and Sn–N bonds. Compound *cis*-**3** crystallizes in the monoclinic space group $P2_1/n$. Bond lengths, bond angles, and torsion angles are given in the Supporting Information. The molecular structure of *cis*-**3** (Figure 2) reveals three interesting features: (a) In contrast to the free ligand **1**, the oxamide crystallizes in a *cis* conformation of point group symmetry C_1, e.g., this compound could be considered as an *anti-Janus head* complex; meaning that the oxamide fragment consists of two different coordination sites pointing towards opposite directions. (b) It is a dinuclear Sn^{II} complex, in which Sn2 is surrounded by two oxygen atoms; fused cycle $SnO_2Cl(O)$, whereas Sn1 is chelated by two nitrogen sites of oxamide ligand; fused cycle $SnN_2Cl(O)$. Each tin atom is further coordinated to a THF molecule resulting in a tetracoordinate *pseudo*-trigonal bipyramidal arrangement. The tin electron lone pairs are probably an orbital with a strong s character.[20] Finally, both tetracoordinate (*pseudo*-tbp) tin atoms are stereogenic centers and, therefore, an isomeric mixture should be expected; however, the reactions yielded only one isomer of 36 isomers (3!•3! = 36), which can be considered as a stereoselective reaction.[21] In the solid state, intermolecular weak interactions forms a dimer of compound *cis*-**2** (Figure 3), where the distance from Sn2 (with less steric demand) to the centroid of the ring C_6 is 3.402 Å [η^6-(2,6-$Me_2C_6H_3$)•••Sn2], which less than the sum of the van der Waals radii (Σr_{vdW} Sn–C = 3.85 Å)[22] and longer than the $Cp^*{}_2Sn$ distance (2.401 Å).[23] Moreover, both oxamide planes are coplanar.



cis-**3**

Scheme 3. Configurational isomer of dinuclear Sn^{II} *cis*-oxamide **3** isolated in solid state.

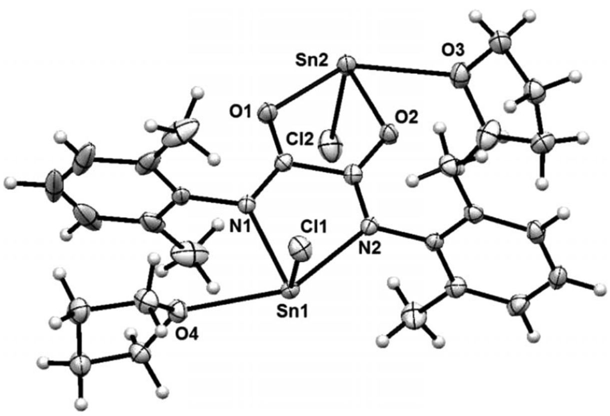


Figure 2. Molecular structure of *cis*-**3**. Thermal ellipsoids are drawn at 50 % probability level (hydrogen atoms are omitted for clarity).

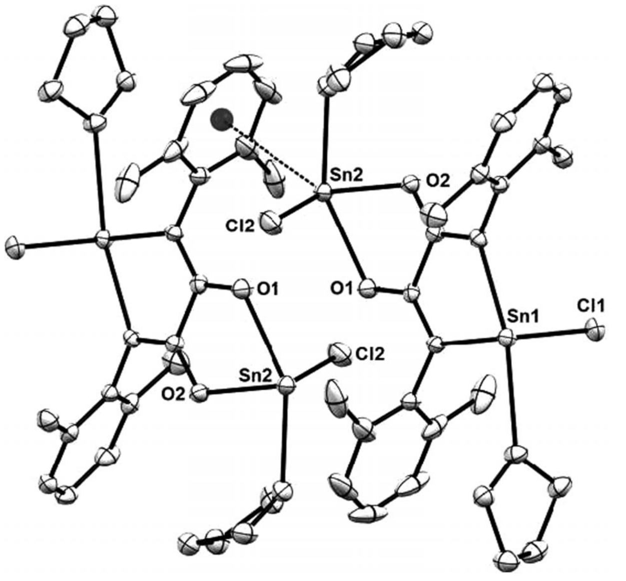


Figure 3. Dimeric aggregate of *cis*-**3**, Sn(2)···C6 (3.402 Å) contact is shown (hydrogen atoms are omitted for clarity).

Finally, in order to better understand the early results, we carried out a series of DFT calculations at the BP86/def2-SVP level. The most relevant geometrical parameters are summarized in Figure 4. The computed bond lengths are in reasonable agreement with the experimental values. The most significant changes are observed in the dative bonds, but it is well-known that these kinds of bonds are shorter in the solid state than in the gas phase.[24] Interestingly, our calculations indicate the *trans* form is slightly more stable as the *cis* isomer by only 0.5 kcal•mol^{-1} (including the zero-point energy). The energy difference is small enough that in principle both isomers could be observed, as the experiments show. However, how could we explain the exclusive *cis* preference?

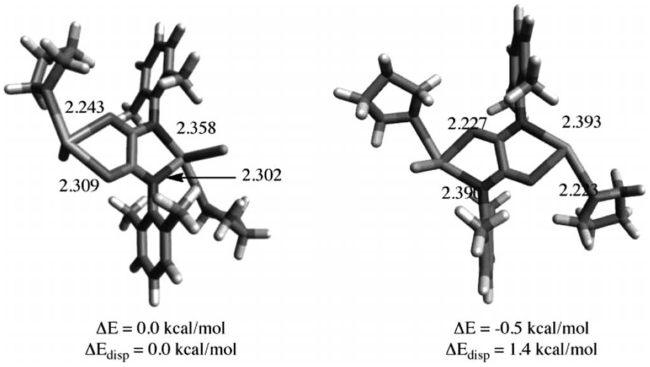


Figure 4. Selected internuclear distances /Å calculated for *cis*-**3** and *trans*-**3**.

Recently, *Grimme* and co-workers discussed in detail the importance of the dispersion energy for the thermodynamic stability of molecules.[25] Given that our ligand has a medium size, the dispersion contribution could be important. Using the implementation of *Grimme*'s DFT-D method for calculating dispersion corrections for DFT functionals[26] we found remarkable effects. Without added dispersion energy, the *trans* isomer is slightly more stable than the *cis* form by only 0.5 kcal•mol^{-1}. Adding the dispersion correction, this energy difference is inverted; now the *anti-Janus head* complex is now more stable than the *trans* form by 1.4 kcal•mol^{-1}. The absolute dispersion to this isomerization is around 2 kcal•mol^{-1}, which is large enough to make a decisive contribution in practice. This indicates that the steric effect is one of the main factors to stabilize the *cis* isomer and the dispersion stabilization occurs for bulky oxamides. Given that dispersion is always attractive, it thermodynamically stabilizes large bulky oxamide complexes compared to small ones.

Finally, the experimentally observed *cis* structure in a condensed phase has one extra can be rationalized taking into account the difference between the dipole moments of *cis* and *trans* forms. Given the local symmetry of *trans*-**2**, it is expected that the dipole moment in *cis*-**2** should be larger. The calculated dipole moments are 1.64 and 0.25 D, respectively. In a condensed phase, the initial aggregation is dominated by the long-range electrostatic interaction. The stronger dipole-dipole interaction of a pair, *cis*-**2**···*cis*-**2**, will drive the system to a preferential *cis* arrangement instead of the structure in the gas phase is quite competitive. This dipole-dipole interactions and the dispersion contribution will stabilize the *cis* form in the solid state.

Conclusions

We described the synthesis, characterization, and X-ray structures of the first example of a dinuclear Sn^{II} *anti-Janus head* complex with a bulky *cis*-oxamide. The calculated geometries are consistent to the X-ray experimental results. Our calculations show that the absolute dispersion to this isomerization is around 2 kcal•mol^{-1}, stabilizing the *anti-Janus head* form. This indicates that the steric effect is one of the main factors to stabilize the *cis* isomer and the dispersion interaction stabilizes bulky oxamides. In the solid state, the dipole-dipole interactions also contribute in the stabilization of the *cis* form.

Experimental Section

Materials and General Methods: All manipulations were performed in a nitrogen atmosphere by using standard Schlenk techniques[27] or inside a glovebox. The THF was dried with Na/benzophenone prior to use in a nitrogen atmosphere. Chemical reagents were commercial available and used as received. Mass spectrum in the EI mode was recorded at 20 eV with a Hewlett-Packard HP 5989 spectrometer and high- resolution mass spectra were obtained by LC/MSD TOF on an Agilent Technologies instrument with APCI as ionization source. IR spectra were obtained with a FT-IR 1600 Perkin-Elmer. The C_6D_6 solvent used for NMR measurements was distilled from Na/K alloy prior to use and $CDCl_3$ used without further purification. Elemental analyses were performed with the Perkin-Elmer Series II CHNS/O analyzer. Melting points were obtained with a Mel-Temp II apparatus. NMR spectra were recorded with a JEOL GSX 270: ^{1}H (270.16 MHz), ^{13}C (67.9 MHz) and JEOL 300 ^{1}H (300 MHz), ^{119}Sn (112.07 MHz): instruments all equipped with multinuclear units. Variable temperature ^{119}Sn-NMR spectra were recorded with a Bruker Avance DPX 400 MHz.

Crystal Structure Determination: The data for **1** was collected at 298(1) K and a suitable crystal of *cis*-**2** was covered with a layer of hydrocarbon oil that was selected and mounted with paratone-N oil on a cryo-loop, and immediately placed in the low-temperature nitrogen stream at 100(2) K. The data for **1** was collected on Siemens P4, with a graphite monochromator. The data for *cis*-**3** was recorded with a Bruker SMART APEX CCD area detector system equipped with a Oxford Cryosystems 700 Series Cryostream cooler, a graphite monochromator, and a Mo-K_α fine-focus sealed tube (λ = 0.71073 Å). Both structures were solved by direct methods using *SHELXS-97*[28] and refined against F^2 on all data by full-matrix least-squares with SHELXL-97.[29]

Crystal Data for 1: Mr = 296.37, orthorhombic, space group *Pcab*, a = 10.016(7) Å, b = 11.773(7) Å, c = 26.129(15) Å, α = 90°, β = 99.142(2)°, γ = 90°, V = 3081(3) Å^3, Z = 2, ρ calcd. = 1.278 g·cm^{-3}, R(reflections) = 0.0735(2699), wR_2(reflections) = 0.1774(10119).

Crystal Data for *cis*-Oxamide 2: Mr = 746.83, monoclinic, space group $P2_1/n$, a = 8.0473(3) Å, b = 11.6507(4) Å, c = 30.9617(11) Å, α = 90°, β = 93.0780(10)°, γ = 90°, V = 2898.68(18) Å^3, Z = 4, ρ calcd. = 1.711 g·cm^{-3}, R(reflections) = 0.0226(5990), wR_2(reflections) = 0.0236(24134).

Crystallographic data (excluding structure factors) for the structures in this paper have been deposited with the Cambridge Crystallographic Data Centre, CCDC, 12 Union Road, Cambridge CB21EZ, UK. Copies of the data can be obtained free of charge on quoting the depository numbers CCDC-804037 for **1** and CCDC-803607 for *cis*-**3** (Fax: +44-1223-336-033; E-Mail: deposit@ccdc.cam.ac.uk, http://www.ccdc.cam.ac.uk).

Theoretical Calculations: All structures were fully optimized at the BP86/def2-SVP level using the Gaussian 03 suite of programs. Every stationary point was characterized as a local minimum on the potential energy surface (PES) by a harmonic (frequency) analysis. Zero-point vibrational energies and thermal corrections to enthalpy were also computed at the unscaled BP86/def2-SVP level. The dispersion were included using the implementation of *Grimme*'s DFT-D method for calculating dispersion corrections for DFT functionals.[26]

Synthesis of *N,N'*-2,6-Dimethylphenyl-oxamide (1): A 250 mL two-neck-flask containing a solution of oxalyl chloride (1.74 mL, 20 mmol) in dry THF (30 mL) in a nitrogen atmosphere was cooled in an ice bath, and a solution 2,6-dimethylaniline of (4.9 mL, 40 mmol) in THF (40 mL) was slowly added with a dropping funnel. This equipment was connected to a saturated NaOH solution for the release of HCl. A white precipitate immediately formed and after 1 h the temperature raised to room temperature and the resulting solid was recovered by filtration, washed with H_2O (10 mL) and dried overnight in air and finally in vacuo. Compound **1** was recrystallized from Et_2O/THF to give suitable single crystals for X-ray diffraction study. Yield 5.07 g (85 %), M.p. 235–237 °C. $C_{18}H_{20}N_2O_2$ (296.37 g·mol^{-1}): calcd. C 72.95, H 6.80, N 9.45 %; found: C 72.71, H 7.26, N 9.41 %. **^{1}H NMR** (270 MHz, $CDCl_3$): δ = 8.89 (s, 2 H, H-2), 7.15 (m, 6 H, H-5 and H-6), 2.27 (s, 12 H, H-7) ppm. **^{13}C NMR** (67.93 MHz, $CDCl_3$): δ = 158.17 (C1), 132.38 (C3), 135.07 (C4), 128.4 (C5 and C6), 18.54 (C7) ppm. **TOF-MS**: [M$^+$] $C_{18}H_{20}N_2O_2$: 297.1598 a.m.u, found: 297.1597 a.m.u (Error = –0.0545 ppm). **IR** (KBr): ṽ = 3294 (N–H), 2962-(C–H), 1665 (C=O), 1497 (C–N), 1220 (C–O), 1039, 802, 672 cm^{-1}.

Synthesis of *N,N'*-2,6-Diisopropylphenyl-oxamide (2): The procedure was similar to the synthesis of oxamide **1**. Yield 7.59 g (93 %), M.p. 301 °C. $C_{26}H_{36}N_2O_2$ (408.52 g·mol^{-1}): calcd. C 76.43, H 8.88, N 6.86 %; found: C 76.53, H 9.99, N 7.06 %. **^{1}H NMR** (400.13 MHz, $CDCl_3$): δ = 8.89 (s, N-H, 2 H), 7.24 (d, 4 H), 7.22 (t, 2 H), 3.08 (sept, *i*Pr, 4 H), 1.24 (d, CH_3, 24 H) ppm. **^{13}C NMR** (100.5 MHz, $CDCl_3$): δ = 159.67 (C=O, 2C), 145.86 (C_q, C2), 129.83 (C_q, C3), 123.83 (C4), 129.0 (C5), 29.17 (C6), 23.66 (C7) 158.17 (C1), 132.38 (C3), 135.07 (C4), 128.4 (C5 and C6), 18.54 (C7) ppm. **TOF-MS**: [M$^+$] $C_{26}H_{36}N_2O_2$: 409.2849 a.m.u, found: 409.2851 a.m.u. (Error = -0.002ppm). **IR** (KBr): ṽ = 3225 (N–H), 2964 (C–H), 1663 (C=O), 1497 (C–N), 1218 (C–O), 903, 801, 728 cm^{-1}.

Synthesis of Complex 3: A solution of n-butyllithium in *n*-hexane (1.6 M, 4.22 mL, 6.75 mmol) was added dropwise at 0 °C to a solution of **1** (0.8 g, 2.7 mmol) in THF (25 mL). After 30 min, a suspension of $SnCl_2$ (1.02 g, 5.4 mmol) in dry THF (15 mL) was added slowly at –78 °C. The reaction mixture was slowly warmed to room temperature and stirred for 16 h. After removal of all volatiles, the residue was extracted with toluene (10 mL) to yield an orange solution of **2**. Yield 0.98 g (52 %), M.p. 98 °C. The product is soluble in benzene, toluene and THF. $C_{18}H_{18}N_2Cl_2O_2Sn_2$·2THF (746.52 g·mol^{-1}): calcd. C 41.83, H 4.55, N 3.75 %; found: C 42.54, H 5.29, N 3.89 %. **^{1}H NMR** (300 MHz, C_6D_6): δ = 7.23 (m, 6 H, H-3 and H-2), 2.20 (s, 12 H, H-1). **^{119}Sn NMR** (112.07 MHz, C_6D_6): δ = –147 ppm. **MS** [EI, *m/z* (%)] = 701 [M$^+$– 3 CH_3 (4)], 533[M$^+$–2Cl, - 2THF (2.5)], 415 [M$^+$–2Cl–Sn–2THF (8)]. **IR** (KBr): ṽ = 2958 (C–H), 1614 (C=O), 1091 (C–O) cm^{-1}.

Note: After the work-up, the *trans*-**3** solution was stored in a freezer at –20 °C for 2 weeks to afford yellow crystals of tin *cis*-oxamide **3** complex (0.31 g, 15.5 %). **IR** (KBr): ṽ = 2958 (C–H), 1614.21 (C=O) and 1568 (C=O), 1095 (C–O) cm^{-1}.

Synthesis of Complex 4: The procedure was similar to the synthesis of the oxamide **3**. Yield 0.88 g (52 %), M.p. 190 °C. $C_{26}H_{34}N_2Cl_2O_2Sn_2$·2THF (858.6 g·mol^{-1}): calcd. C 47.55, H 5.82, N 3.26 %; found: C 47.35, H 5.70, N 3.59 %. **^{1}H NMR** (300 MHz, C_6D_6): δ = 7.23 (m, 6 H), 3.60 (sept, *i*Pr, 4 H), 1.25 (s, CH_3, 24 H) ppm. **^{119}Sn NMR** (112.07 MHz, C_6D_6): δ = –146.05 ppm. **MS** [EI, *m/z* (%)] = 757 [M$^+$–Cl, (1)], 647 [M$^+$–2Cl, (1)], 525.3 [M$^+$–2Cl-Sn], (36)].

Supporting Information (see footnote on the first page of this article): Selected bond lengths and angles and details of the X-ray crystallographic analysis for **1** and *cis*-**3**. ^{1}H, ^{13}C, ^{119}Sn NMR spectra for ligands and tin(II) complexes.

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Dinuclear Tin(II) Complex of a Bulky *cis*-Oxamide: Synthesis, Characterization, Crystal Structure, and DFT Studies