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[{2,6-(Me_2NCH_2)$_2$$C_6H_3$}Sn(μ-OH)W(CO)$_5$]$_2$: A Transition-Metal-Coordinated Organotin(II) Hydroxide

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Dedicated to Professor Herbert Roesky on the occasion of his 75th birthday

Keywords: Tin / Tungsten / X-ray diffraction / Density functional calculations / Organotin hydroxides / Hydrogen bonds

The synthesis of the intramolecularly coordinated pentacarbonyltungsten–organostannylene complexes {2,6-(Me_2NCH_2)$_2$$C_6H_3$}Sn(H)W(CO)$_5$ (**2**) and [{2,6-(Me_2NCH_2)$_2$-C_6H_3}Sn(μ-OH)W(CO)$_5$]$_2$ (**3**) and the solid-state molecular structure of **3**, as determined by single-crystal X-ray diffraction analysis, are reported. Compound **3** is characterized by hydroxido bridges [Sn–O 2.068(5), 2.231(5) Å], an intramolecular N→Sn coordination bond [2.501(7) Å] and an intramolecular O–H···N hydrogen bond [O···N 2.715(8) Å].

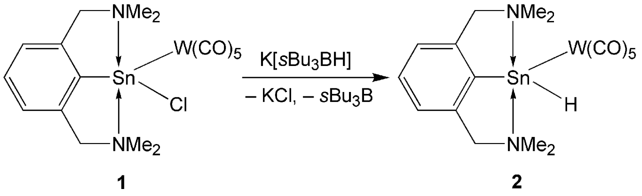
Introduction

While investigating the chemistry of low-valent group 14 elements[1–6] in general, and tin(II) chemistry in particular, we became aware of only one inorganic tin(II) and one organotin(II) hydroxide, namely the salt-like trinuclear oxidotin cluster $Sn_3(OH)_4(NO_3)_2$[7] and the (Ar′SnOH)$_2$ [Ar′ = 2,6-(2,6-*i*Pr$_2$$C_6H_3$)$_2$$C_6H_3$] compound reported by Power et al.[8] Moreover, the combined oxidation and hydrolysis of [4-*t*Bu-2,6-{(O*i*Pr)P(O)}$_2$$C_6H_2$SnCr(CO)$_5$]$_2$ provided the corresponding transition-metal-complexed organotin(II) hydroxide [4-*t*Bu-2,6-{(O*i*Pr)P(O)}$_2$$C_6H_2$Sn(OH)Cr(CO)$_5$]$_2$.[9] Details of the latter will be reported in a forthcoming paper.

Based on our findings that, in solution and with the evolution of hydrogen,[10] the hydrido-substituted organostannylene transition metal complexes [4-*t*Bu-2,6-{(OR)$_2$P(O)}$_2$$C_6H_2$Sn(H)M] [M = Cr(CO)$_5$, W(CO)$_5$, Fe(CO)$_4$; R = Et, *i*Pr] undergo tin–tin bond formation to give the corresponding ditin compounds [4-*t*Bu-2,6-{(OR)$_2$P(O)}$_2$-C_6H_2SnM]$_2$,[9,11] we attempted the synthesis of the related complex [{2,6-(Me_2NCH_2)$_2$$C_6H_3$}SnW(CO)$_5$]$_2$ that contains (dimethylamino)methyl (Me_2NCH_2) instead of the phosphonic ester (P(O)(OR)$_2$) substituents. However, this goal was not achieved. Instead, we learned that [{2,6-(Me_2NCH_2)$_2$$C_6H_3$}Sn(H)W(CO)$_5$] (**2**) easily undergoes hydrolysis to give [{2,6-(Me_2NCH_2)$_2$$C_6H_3$}Sn(μ-OH)W(CO)$_5$]$_2$ (**3**), a rare example of a transition-metal-coordinated organotin(II) hydroxide. While this work was underway, Roesky and co-workers reported a rational synthesis for the tetracarbonyliron-bound organotin hydroxide [(OC)$_4$FeSn(OH){N(Ar)C(Me)}$_2$CH].[12]

Results and Discussion

The reaction of the intramolecularly coordinated chlorido(organo)stannylene–pentacarbonyltungsten complex [{2,6-(Me_2NCH_2)$_2$$C_6H_3$}Sn(Cl)W(CO)$_5$] (**1**)[13] with K[*s*Bu$_3$BH] gave the corresponding organotin(II) hydride complex **2** (Scheme 1) as a pale yellow air-and moisture-sensitive solid material that is very soluble in organic solvents such as benzene or THF.



Scheme 1.

The ^1H NMR spectrum (300.13 MHz, C_6D_6) of the organotin(II) hydride complex **2** showed two equally intense singlets at δ = 1.93 and 2.23 ppm and an AX spin system at δ = 2.71 and 3.54 ppm. These resonances are assigned to the diastereotopic NCH_3 and NCH_2 protons and indicate

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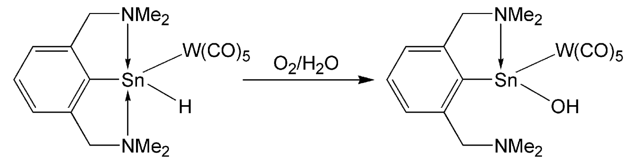
that the intramolecular N→Sn coordination is inert on the ^{1}H NMR time scale. Furthermore, there is a sharp signal at δ = 9.29 ppm with a $^{1}J(^{1}H\text{-}^{119}Sn)$ coupling constant of 1302 Hz that is assigned to the Sn*H* proton. The $^{1}J(^{1}H\text{-}^{119}Sn)$ coupling constant in compound **2** is slightly larger than those reported for the related organotin(II) hydrides [4-*t*Bu-2,6-$\{(OiPr)_2P(O)\}_2C_6H_2$]Sn(H)W(CO)$_5$ (1198 Hz)[14] and [{2,6-(MeOCH$_2$)$_2$C$_6$H$_3$}Sn(H)W(CO)$_5$] (1118 Hz).[15] The ^{119}Sn NMR spectrum of **2** displayed a sharp resonance at δ = 199 ppm with a $^{1}J(^{119}Sn\text{-}^{183}W)$ constant of 870 Hz. This value is smaller than the corresponding couplings in the chlorido-substituted derivative **1** (1186 Hz) and in the oxygen-coordinated complexes [2,6-(ROCH$_2$)$_2$C$_6$H$_3$Sn(X)-W(CO)$_5$] [R = Me, X = Cl (1245 Hz); R = *t*Bu, X = Cl (1289 Hz); R = Me, X = H (1242 Hz)][15] and [4-*t*Bu-2,6-{(O-*i*Pr)$_2$P(O)}$_2$C$_6$H$_2$]Sn(Cl)W(CO)$_5$ (1366 Hz).[14] No single crystals of compound **2** suitable for X-ray diffraction analysis could be obtained, and consequently the structure of **2** in the solid state remains unknown. However, analogous to [4-*t*Bu-2,6-{(O*i*Pr)$_2$P(O)}$_2$C$_6$H$_2$]Sn(H)Cr(CO)$_5$, the structure of which has been shown to be monomeric,[9] compound **2** is likely to exhibit a similar monomeric structure.

The reaction of a toluene solution of the hydrido-substituted complex **2** with water exclusively provided the hydroxido-substituted compound **3** as a crystalline material that is sparingly soluble in CH$_2$Cl$_2$ and thf (Scheme 2).



3

Scheme 2.

The ^{1}H NMR spectrum (300.13 MHz, C$_6$D$_6$) of complex **3** showed one broad signal for NC*H*$_3$ at δ = 2.50 ppm, and an AB spin system for NC*H*$_2$ at δ = 3.68 ppm. The ^{119}Sn NMR spectrum of **3** displayed a sharp resonance at δ = 79 ppm that is shifted to a lower frequency when compared to the corresponding signal in the spectrum of **2** (δ = 199 ppm). The IR spectrum of **3** shows an absorption band at 3055 cm^{-1} corresponding to ν(O–H), and two bands at 2063 and 1940 cm^{-1}, which are diagnostic positions for ν(C–O).

The molecular structure of compound **3** is shown in Figure 1, selected interatomic distances and angles are given in Table 1, and a comparison of relevant data with related derivatives (Scheme 3) is made in Table 2.

Compound **3** is a centrosymmetric μ-hydroxido-bridged dimer. The tin atom is five-coordinate and shows a strongly distorted trigonal-bipyramidal configuration with the N(1) and O(1A) atoms occupying the axial positions and the C(1), O(1A), and W(1) atoms occupying the equatorial positions. The N(1)–Sn(1) distance of 2.501(7) Å is slightly shorter than the N–Sn distances found in the corre-

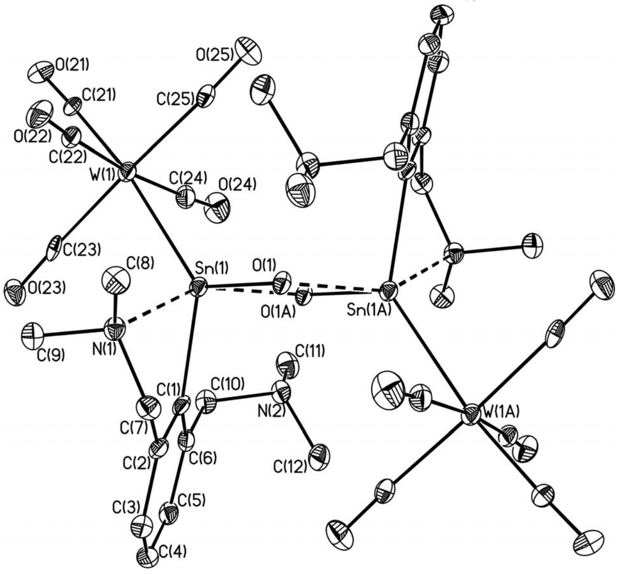
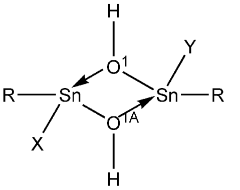


Figure 1. Molecular structure of compound **3**. Thermal ellipsoids are shown at the 30% probability level.

Table 1. Selected interatomic distances [Å], angles [°] and torsion angles [°] for compound **3** as measured by single-crystal X-ray diffraction analysis and from the optimized structure **3a** obtained by DFT calculations with different basis sets.

	3 X-ray	**3a** LANL2DZ	**3a** def2-TZVP
C(1)–Sn(1)	2.176(7)	2.181	2.217
N(1)–Sn(1)	2.501(7)	2.492	2.601
O(1)–Sn(1)	2.068(5)	2.048	2.125
O(1A)–Sn(1)	2.231(5)	2.181	2.261
W(1)–Sn(1)	2.7644(6)	2.869	2.852
N(2)···O(1A)	2.715(8)	2.704	2.830
O(1)···O(1A)	2.616(9)	2.511	2.623
Sn(1)···Sn(1A)	3.4160(8)	3.404	3.515
C(1)–Sn(1)–N(1)	74.0(3)	74.0	72.6
C(1)–Sn(1)–O(1)	102.1(2)	107.5	102.7
C(1)–Sn(1)–O(1A)	90.5(2)	87.3	90.8
C(1)–Sn(1)–W(1)	130.3(2)	130.3	132.8
N(1)–Sn(1)–O(1)	81.3(2)	84.7	82.2
N(1)–Sn(1)–O(1A)	148.1(2)	144.9	146.7
N(1)–Sn(1)–W(1)	105.8(1)	106.7	106.1
O(1)–Sn(1)–O(1A)	74.8(2)	72.8	73.4
O(1)–Sn(1)–W(1)	127.4(1)	122.1	125.2
O(1A)–Sn(1)–W(1)	105.4(1)	108.0	106.4
Sn(1)–O(1)–Sn(1A)	105.2(2)	107.2	106.6
W(1)–Sn(1)–Sn(1A)–W(1A)	180.0	180.0	180.0
W(1)–Sn(1)–Sn(1A)–C(1)	30.5(2)	30.9	27.9



A: R = 2,6-(2,6-*i*Pr$_2$C$_6$H$_3$)$_2$C$_6$H$_3$, X = Y = lone pair
B: R = 2,6-(2,4,6-*i*Pr$_3$C$_6$H$_2$)$_2$C$_6$H$_3$, X = lone pair, Y = W(CO)$_5$
C: R = 4-*t*Bu-2,6-[(RO)$_2$P(O)]$_2$C$_6$H$_2$, X = Y = Cr(CO)$_5$
3: R = 2,6-(Me$_2$NCH$_2$)$_2$C$_6$H$_3$, X = Y = W(CO)$_5$

Scheme 3. Schematic representation of known dimeric organotin(II) hydroxides.

Table 2. Comparison of structural data for known dimeric organotin(II) hydroxides.

	3	A[8]	B[17]	C[9]
Sn(1)–O(1)	2.068(5)	2.140(2)	2.140(2)	2.118(2)
Sn(1)–O(1A)	2.231(5)	2.149(2)	2.224(5)	2.132(2)
Sn(1)–Sn(1A)	3.416(1)	3.460(2)	3.475(1)	3.556(2)
O(1)–O(1A)	2.616(1)	2.527(3)	2.538(5)	2.329(1)
O(1)–Sn(1)–O(1A)	74.8(2)	72.27(6)	73.0(3)/ 69.9(3)	66.4(1)
Sn(1)–O(1)–Sn(1)	105.2(2)	107.73(6)	105.6(2)	113.6(1)

sponding organostannylene [2,6-$(Me_2NCH_2)_2C_6H_3SnCl$] [2.525(8), 2.602(8) Å][16] and the pentacarbonyltungsten complex [{2,6-$(Me_2NCH_2)_2C_6H_3$}Sn(Cl)W$(CO)_5$] [2.543(3), 2.5526(3) Å],[13]but is longer than the corresponding distance in the aqua complex [{2,6-$(Me_2NCH_2)_2$-C_6H_3}Sn(OH)W$(CO)_5$]$^+$$(CB_{11}H_{12})^-$ [2.264(2) Å].[13] Similar to the latter compound, in which the second nitrogen atom does not coordinate to the tin ion but is involved in a hydrogen bond to the water oxygen atom [N···O 2.66(3) Å], the N(2) atom in compound **3** is involved in a hydrogen bond to the hydroxido oxygen atom O(1A) with an N(2)···O(1A) distance of 2.714(1) Å. The μ-hydroxido bridge is nonsymmetric with Sn(1)–O(1) and Sn(1A)–O(1) distances of 2.068(5) and 2.231(5) Å, respectively. In the related compounds **A**–**C**,[8,9,17] the μ-hydroxido bridges are less nonsymmetric (Table 2). All these Sn–O distances are, however, longer than the Sn(1)–O(5) distance of 2.001(3) Å in [$(OC)_4$-FeSn(OH){N(Ar)C(Me)}$_2$CH];[12] however, this compound is monomeric, and the tin atom is four-coordinate.

DFT Calculations

DFT calculations for compound **3** gave the geometry-optimized structure **3a**. These calculations also give an insight into the bonding situation and elucidate the influence of the (dimethylamino)methyl substituents, which have the potential to coordinate intramolecularly, on the Sn–O→Sn bridge. The presence of a strong O–H···N hydrogen bond in compound **3** raises the question as to whether or not the nitrogen atom of the (dimethylamino)methyl group is basic enough to deprotonate the hydroxido bridge to give the zwitterionic oxido-bridged compound **3b** (Scheme 4).

The rather simple LANL2DZ basis set reproduces the geometrical parameters of compound **3** quite well in comparison to the more elaborate def2-TZVP basis set that predicts all bond lengths to be slightly longer than the experimentally determined values (Table 1). This tendency is often encountered for triple-zeta basis sets.[18] The structure of **3a** can be optimised to give a structure located at a stable energy minimum, whereas the structure **3b** is computationally not stable and converts in silico to **3a**.

By constraining the artificial N–H bond, a model structure for **3b** was obtained. The key geometric parameters for **3b** are summarised in Table 3. By elongation of the O–H

Table 3. Selected interatomic distances [Å], angles [°] and torsion angles [°] for the calculated structures **3b** and **3c** (RB3LYP/LANL2DZ).

	3b bis(μ-oxido)	**3c** (μ-hydroxido)(μ-oxido)
C(1)–Sn(1)	2.221	2.202, 2.190
N(1)–Sn(1)	2.629	2.577, 2.508
O(1)–Sn(1)	2.097	2.049, 2.006
O(1A)–Sn(1)	2.014	2.198, 2.088
W(1)–Sn(1)	2.888	2.879, 2.880
N(2)···O(1A)	2.730	2.703, 2.733
O(1)···O(1A)	2.586	2.555
Sn(1)···Sn(1A)	3.197	3.297
O(1)–Sn(1)–O(1A)	77.9	76.3
Sn(1)–O(1)–Sn(1A)	102.1	107.2

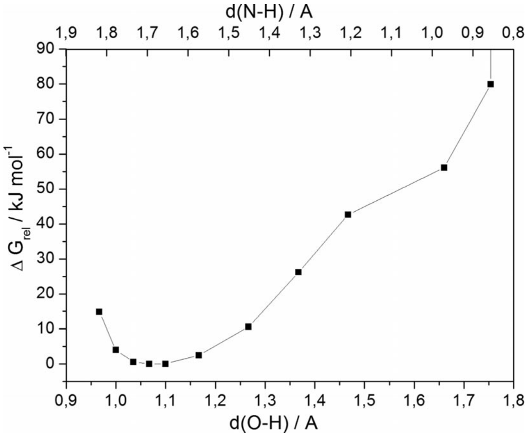
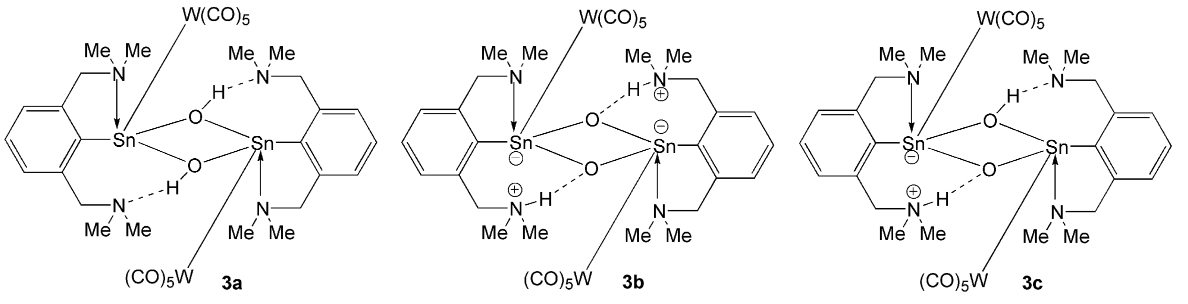


Figure 2. Energy profile for the O–H distance in the computational model of compound **3** (B3LYP/LANL2DZ).



Scheme 4. Schematic representation of the calculated structure **3a**, of the doubly deprotonated bis(μ-oxido) structure **3b**, and of the singly deprotonated (μ-hydroxido)(μ-oxido) structure **3c**.

bond in the direction of the N donor, an energy profile for the "artificial deprotonation" was derived (Figure 2), which clearly shows that the bis(μ-hydroxido) dimer is favored over the bis(μ-oxido) dimer by approximately 80 kJ/mol. As deprotonation is not expected to occur simultaneously, a further model compound, a model for the (μ-hydroxido)(μ-oxido) dimer **3c**, was calculated. In terms of energy, this theoretical model resides between the endpoints **3a** and **3b**. The influence of the hydroxido/oxido bridges in **3a**, **3b**, and **3c** on the coordination sphere of the tin ions has to be highlighted (Table 3). The N–Sn distance increases in the order **3a** < **3c** < **3b**, whereas the O–Sn distances in the Sn_2O_2 four-membered ring are longest in **3a**. However, the Sn···Sn distance of 3.197 Å in **3b** is shorter than the 3.404 Å calculated for **3a**.

Conclusions

In this work we report the pentacarbonyltungsten-complexed organotin(II) hydroxide **3** and show that, as an alternative to bulky substituents, N,C,N-coordinating pincer-type ligands can stabilize such compounds by both N→Sn coordination and O–H···N hydrogen bonds. Interestingly, out of the five reported organotin(II) hydroxides (transition-metal-complexed or not) only the tetracarbonyliron complex $[(OC)_4FeSn(OH)\{N(Ar)C(Me)\}_2CH]$[12] is monomeric and lacks intermolecular μ-hydroxido bridges. Apparently, in this compound the diketiminato ligand reduces the Lewis acidity of the tin ion to a greater extent than the N,C,N- and O,C,O-coordinating ligands do in compounds **3** and **C**. Moreover, based on DFT calculations, it appears that compounds such as $(cyclo\text{-}RSnO_2SnR)^{2-}(NHR_3^+)_2$ are not stable.

Experimental Section

General Methods: Solvents were dried according to standard methods and distilled prior to use. All moisture- and air-sensitive reactions were carried out under argon with standard Schlenk techniques. The synthesis of the chlorido-substituted organostannylene complex **1** is reported elsewhere.[13] Elemental analyses were performed with a LECO CHNS-932 analyzer. The ^1H, ^{13}C, and ^{119}Sn NMR spectra were acquired with Bruker DRX400 and DPX300 FT NMR spectrometers with broad-band decoupling of ^{119}Sn at 149.21 or 111.92 MHz and of ^{13}C at 100.61 MHz, with external and internal deuterium lock. The ^1H, ^{13}C, and ^{119}Sn NMR chemical shifts (δ) are given in ppm and referenced to external Me_4Sn (^{119}Sn), Me_4Si (^{13}C, ^1H) standards, The FAB mass spectra were measured with a Jeol JMS-SX 102a spectrometer. The IR spectra were recorded with a Perkin–Elmer 684 spectrometer.

Synthesis of $[\{2,6\text{-}(Me_2NCH_2)_2C_6H_3\}Sn(H)W(CO)_5]$ (2): A 1 M thf solution (0.3 mL) of $K[sBu_3BH]$ (0.3 mmol) was added to a solution of compound **1** (0.22 g, 0.3 mmol) in thf (20 mL) at –78 °C. The reaction mixture was stirred at this temperature for 3 h and then warmed to room temperature. Evaporation of the solvent gave a residue that was suspended in toluene (20 mL). The suspension was filtered, and the filtrate was stripped of all volatiles to give a solid material. The latter was washed with *n*-hexane to give **1** as a pale yellow solid. Yield: 0.13 g (68%); m.p. 153 °C (decomp.). $C_{17}H_{20}N_2O_5SnW$ (634.9): calcd. C 32.16, H 3.18; found C 31.9, H 3.03. FAB-MS: m/z = 636 $[M + H]^+$, 634 $[M - H]^+$. ^1H NMR (C_6D_6, 300.13 MHz): δ = 1.93 (s, 6 H, CH_3), 2.23 (s, 6 H, CH_3), 2.71 [AX system, $^2J(^1H,^1H)$ = 14.3 Hz, 2 H, CH_2], 3.54 [AX system, $^2J(^1H\text{-}^1H)$ = 14.3 Hz, 2 H, CH_2], 6.74 (d, 2 H, Ar), 7.06 (t, 1 H, Ar), 9.29 [s, $^1J(^1H\text{-}^{119}Sn)$ = 1302, $^2J(^1H,^{182}W)$ = 15.3 Hz, 1 H] ppm. ^{13}C NMR (C_6D_6, 75.63 MHz): δ = 47.2 (CH_3), 47.7 (CH_3), 65.1 (CH_2), 124.7 [C(3,5)], 128.6 [C(4)], 143.6 [(C(2,6)], 150.4 [(C(1)], 200.9 [CO_{eq}, $^2J(^{119}Sn\text{-}^{13}C)$ = 53, $^1J(^{183}W\text{-}^{13}C)$ = 123 Hz], 202.4 (CO_{ax}) ppm. ^{119}Sn NMR (C_6D_6, 111.95 MHz): δ = 199.0 [$^1J(^{182}W, ^{119}Sn)$ = 870 Hz)] ppm.

Synthesis of $[\{2,6\text{-}(Me_2NCH_2)_2C_6H_3\}Sn(\mu\text{-}OH)W(CO)_5]_2$ (3): Several drops of water were added to a solution of **2** (0.1 g, 0.16 mmol) in toluene (5 mL). After stirring of the reaction mixture for 5 h, the toluene was evaporated in vacuo to give a white solid that was washed with *n*-hexane. Recrystallization of this solid from benzene provided 0.80 g (78% yield) of crystalline **3**; m.p. 152–156 °C. $C_{34}H_{40}N_4O_{12}Sn_2W_2$ (1301.93): calcd. C 31.37, H 3.10; found 31.20 C, 3.24 H. ^1H NMR ($CDCl_3$, 300.13 MHz): δ = 2.50 (br. s, 12 H, CH_3), 3,68 [AB system, $^2J(^1H,^1H)$ = 13.6 Hz, 4 H, CH_2], 7.07 (d, 1 H, Ar), 7.09 (d, 1 H, Ar), 7.25 (t, 1 H, Ar) ppm. ^{13}C NMR ($CDCl_3$, 75.63 MHz): δ = 45.3 (CH_3), 65.0 (CH_2), 125.1 [C(3,5)], 129.3 [C(4)], 143.9 [(C(2,6)], 154.9 [C(1)], 199.5 (CO_{eq}), 200.4 (CO_{ax}) ppm. ^{119}Sn NMR ($CDCl_3$, 111.95 MHz): δ = 79 ppm. IR (CH_2Cl_2): $\tilde{\nu}$ = 1940 (vs, CO), 2063 (vs, CO), 3055 (s, OH).

Computational Details: Calculations were performed with the program suites Gaussian 03[19] and Turbomole.[20] All geometries were optimized with the B3LYP hybrid DFT functional[21] and the Los Alamos National Laboratory double-zeta basis set (LANL2DZ). Structure **3a** was also optimized with the BP86 pure functional[21a,22] and the Ahlrichs def2-TZVP basis set,[23] which includes effective core potentials for Sn and W. Tight conversion criteria were applied. Each stationary point has been characterized with frequency analysis and shows the correct number of negative eigenvalues (zero for a local minimum and one for a transition state).

Crystallography: Intensity data for the colorless crystal of **3** were collected with a Nonius KappaCCD diffractometer with graphite-monochromated Mo-K_α radiation at 173 K. The data collection covered almost the whole sphere of reciprocal space with 4 sets at different κ-angles and 377 frames collected by ω rotation (Δ/ω = 1°) at two times (10 s per frame). The crystal-to-detector distance was 3.4 cm. Crystal decay was monitored by repeating the initial frames at the end of data collection. Analysis of the duplicate reflections indicated that there was no crystal decay over the timescale of the data collection. The structure was solved by direct methods with SHELXS97[24] and from successive difference Fourier syntheses. Refinement was applied with full-matrix least-squares methods with SHELXL97.[25] All H atoms were located in the difference Fourier maps, and their positions were isotropically refined with U_{iso} constrained to be 1.2 times the U_{eq} of the carrier C atom for non-methyl hydrogen atoms and 1.5 times the U_{eq} of the carrier C atom for all methyl hydrogen atoms. Atomic scattering factors for neutral atoms and real and imaginary dispersion terms were taken from the International Tables for X-ray Crystallography.[26] The figures were created with SHELXTL.[27] Crystallographic data are given in Table 4 and selected bond lengths and angles in Table 1. CCDC-789208 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Table 4. Crystal data and structure refinement details for compound **3**.

Empirical formula	$C_{34}H_{38}N_4O_{12}Sn_2W_2$
Formula mass	1299.76
Temperature [K]	173(1)
Wavelength [Å]	0.71073
Crystal system, space group	triclinic, $P\bar{1}$
Unit cell dimensions	
a [Å]	9.8601(11)
b [Å]	11.0339(14)
c [Å]	11.1201(8)
α (°)	98.340(8)
β (°)	112.955(6)
γ (°)	107.816(5)
Volume [Å^3]	1011.2(2)
Z	1
Density (calcd.) [Mg/m^3]	2.134
Absorption coefficient μ [mm^{-1}]	6.952
F(000)	614
Crystal size [mm]	0.25 × 0.18 × 0.10
θ range for data collection [°]	3.32–27.47
Index ranges	$-12 \le h \le 12$ $-14 \le h \le 13$ $-14 \le h \le 12$
Reflections collected/unique [R(int) = 0.055]	12065/4568
Reflections observed [$I > 2\sigma(I)$]	3017
Completeness to 2θ = 54.96°	98.7%
Absorption correction	semi-empirical
Max./min. transmission	0.543/0.270
Refinement method	full-matrix least squares on F^2
Data/restraints/parameters	4568/0/233
Goodness-of-fit on F^2	0.939
Final R indices [$I > 2\sigma(I)$]	R_1 = 0.0372, $wR2$ = 0.0922
R indices (all data)	R_1 = 0.0631, $wR2$ = 0.0957
Max. shift/esd	< 0.001
Largest diff. peak/hole [e Å^{-3}]	2.678/–2.012

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