

Preparation and Full Characterization of a Tetrahydride-bis(stannyl)-osmium(VI) Derivative

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Supporting Information Available: Tables of positional and displacement parameters, crystallographic data, and bond lengths and angles for the complex 2. Computational details.

Table 1. Crystal data and structure refinement for 2.

Identification code	2
Empirical formula	C ₅₄ H ₇₆ Os P ₂ Sn ₂
Formula weight	1214.67
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, C 1 2/c 1
Unit cell dimensions	a = 12.7173(13) Å alpha = 90 deg. b = 20.0547(13) Å beta = 107.547(2) deg. c = 20.9584(17) Å gamma = 90 deg.
Volume	5096.5(7) Å ³
Z, Calculated density	4, 1.583 Mg/m ³
Absorption coefficient	3.556 mm ⁻¹
F(000)	2424
Crystal size	0.60 x 0.20 x 0.10 mm
Theta range for data collection	1.96 to 28.70 deg.
Limiting indices	-17<=h<=13, -27<=k<=26, -27<=l<=27
Reflections collected / unique	16921 / 6092 [R(int) = 0.0715]
Completeness to theta = 28.70	92.7 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.862 and 0.474
Refinement method	Full-matrix least-squares on F ²

Data / restraints / parameters	6092 / 0 / 281
Goodness-of-fit on F ²	0.825
Final R indices [I>2sigma(I)]	R1 = 0.0374, wR2 = 0.0606
R indices (all data)	R1 = 0.0524, wR2 = 0.0641
Largest diff. peak and hole	1.319 and -1.454 e.A ⁻³

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 2. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
Os	10000	9100(1)	2500	24(1)
Sn	8694(1)	8251(1)	2906(1)	27(1)
P	11380(1)	9784(1)	3273(1)	28(1)
C(1)	11229(4)	9860(2)	4123(2)	33(1)
C(2)	11202(4)	9171(2)	4445(2)	45(1)
C(3)	10294(4)	10291(2)	4189(2)	42(1)
C(4)	11436(4)	10677(2)	3054(2)	30(1)
C(5)	12158(4)	11138(2)	3590(2)	41(1)
C(6)	11698(4)	10785(2)	2388(2)	40(1)
C(7)	12802(4)	9428(2)	3495(2)	34(1)
C(8)	13647(4)	9635(2)	4166(2)	49(1)
C(9)	13355(4)	9509(2)	2951(2)	43(1)
C(10)	7900(4)	8577(2)	3639(2)	30(1)
C(11)	7525(4)	8095(2)	4002(2)	41(1)
C(12)	6953(4)	8262(3)	4442(2)	45(1)
C(13)	6755(4)	8916(3)	4548(2)	48(1)
C(14)	7120(4)	9399(3)	4214(2)	49(1)
C(15)	7688(4)	9233(2)	3769(2)	40(1)
C(16)	7379(4)	7795(2)	2123(2)	30(1)
C(17)	7564(4)	7506(2)	1569(2)	33(1)
C(18)	6742(4)	7201(2)	1068(2)	40(1)
C(19)	5692(4)	7184(2)	1112(2)	40(1)
C(20)	5466(4)	7466(2)	1649(2)	43(1)
C(21)	6298(4)	7763(2)	2151(2)	37(1)
C(22)	9514(3)	7379(2)	3448(2)	29(1)
C(23)	9505(4)	6779(2)	3121(2)	35(1)
C(24)	10056(4)	6229(2)	3453(2)	41(1)
C(25)	10602(4)	6256(2)	4123(3)	48(1)
C(26)	10593(4)	6844(2)	4467(2)	48(1)
C(27)	10064(4)	7396(2)	4122(2)	39(1)

Table 3. Bond lengths [Å] and angles [deg] for 2.

Os-P	2.4234(11)
Os-P#1	2.4234(11)
Os-Sn	2.6897(3)
Os-Sn#1	2.6897(3)
Os-H(01)	1.35(3)
Os-H(02)	1.35(3)
Sn-C(16)	2.162(4)
Sn-C(10)	2.176(4)
Sn-C(22)	2.174(4)
P-C(1)	1.854(4)
P-C(4)	1.856(4)
P-C(7)	1.868(4)
C(1)-C(3)	1.511(6)
C(1)-C(2)	1.542(6)
C(1)-H(1)	1.0000
C(2)-H(2A)	0.9800
C(2)-H(2B)	0.9800
C(2)-H(2C)	0.9800
C(3)-H(3A)	0.9800
C(3)-H(3B)	0.9800
C(3)-H(3C)	0.9800
C(4)-C(5)	1.530(6)
C(4)-C(6)	1.543(6)
C(4)-H(4)	1.0000
C(5)-H(5A)	0.9800
C(5)-H(5B)	0.9800
C(5)-H(5C)	0.9800
C(6)-H(6A)	0.9800
C(6)-H(6B)	0.9800
C(6)-H(6C)	0.9800
C(7)-C(9)	1.516(6)
C(7)-C(8)	1.547(6)
C(7)-H(7)	1.0000
C(8)-H(8A)	0.9800
C(8)-H(8B)	0.9800
C(8)-H(8C)	0.9800
C(9)-H(9A)	0.9800
C(9)-H(9B)	0.9800
C(9)-H(9C)	0.9800
C(10)-C(15)	1.388(5)
C(10)-C(11)	1.401(6)
C(11)-C(12)	1.377(6)
C(11)-H(11)	0.9500
C(12)-C(13)	1.367(6)
C(12)-H(12)	0.9500
C(13)-C(14)	1.356(7)
C(13)-H(13)	0.9500
C(14)-C(15)	1.382(6)
C(14)-H(14)	0.9500
C(15)-H(15)	0.9500
C(16)-C(17)	1.381(5)
C(16)-C(21)	1.395(6)
C(17)-C(18)	1.381(6)
C(17)-H(17)	0.9500
C(18)-C(19)	1.367(6)
C(18)-H(18)	0.9500
C(19)-C(20)	1.365(6)

C(19)-H(19)	0.9500
C(20)-C(21)	1.382(6)
C(20)-H(20)	0.9500
C(21)-H(21)	0.9500
C(22)-C(27)	1.374(6)
C(22)-C(23)	1.382(5)
C(23)-C(24)	1.378(6)
C(23)-H(23)	0.9500
C(24)-C(25)	1.368(6)
C(24)-H(24)	0.9500
C(25)-C(26)	1.384(6)
C(25)-H(25)	0.9500
C(26)-C(27)	1.380(6)
C(26)-H(26)	0.9500
C(27)-H(27)	0.9500
P-Os-P#1	111.10(5)
P-Os-Sn	122.56(3)
P#1-Os-Sn	100.24(3)
P-Os-Sn#1	100.24(3)
P#1-Os-Sn#1	122.56(3)
Sn-Os-Sn#1	101.473(15)
P-Os-H(01)	78.2(15)
P#1-Os-H(01)	83.6(15)
Sn-Os-H(01)	58.5(14)
Sn#1-Os-H(01)	151.2(14)
P-Os-H(02)	77.5(15)
P#1-Os-H(02)	165.8(15)
Sn-Os-H(02)	65.6(15)
Sn#1-Os-H(02)	64.5(14)
H(01)-Os-H(02)	87.3(19)
C(16)-Sn-C(10)	104.20(16)
C(16)-Sn-C(22)	101.24(15)
C(10)-Sn-C(22)	97.13(15)
C(16)-Sn-Os	115.97(11)
C(10)-Sn-Os	119.72(11)
C(22)-Sn-Os	115.49(10)
C(1)-P-C(4)	100.49(19)
C(1)-P-C(7)	99.5(2)
C(4)-P-C(7)	108.9(2)
C(1)-P-Os	115.15(15)
C(4)-P-Os	117.00(14)
C(7)-P-Os	113.70(14)
C(3)-C(1)-C(2)	110.5(4)
C(3)-C(1)-P	116.6(3)
C(2)-C(1)-P	111.7(3)
C(3)-C(1)-H(1)	105.7
C(2)-C(1)-H(1)	105.7
P-C(1)-H(1)	105.7
C(1)-C(2)-H(2A)	109.5
C(1)-C(2)-H(2B)	109.5
H(2A)-C(2)-H(2B)	109.5
C(1)-C(2)-H(2C)	109.5
H(2A)-C(2)-H(2C)	109.5
H(2B)-C(2)-H(2C)	109.5
C(1)-C(3)-H(3A)	109.5
C(1)-C(3)-H(3B)	109.5
H(3A)-C(3)-H(3B)	109.5
C(1)-C(3)-H(3C)	109.5
H(3A)-C(3)-H(3C)	109.5
H(3B)-C(3)-H(3C)	109.5

C(5)-C(4)-C(6)	109.5(4)
C(5)-C(4)-P	117.4(3)
C(6)-C(4)-P	113.3(3)
C(5)-C(4)-H(4)	105.2
C(6)-C(4)-H(4)	105.2
P-C(4)-H(4)	105.2
C(4)-C(5)-H(5A)	109.5
C(4)-C(5)-H(5B)	109.5
H(5A)-C(5)-H(5B)	109.5
C(4)-C(5)-H(5C)	109.5
H(5A)-C(5)-H(5C)	109.5
H(5B)-C(5)-H(5C)	109.5
C(4)-C(6)-H(6A)	109.5
C(4)-C(6)-H(6B)	109.5
H(6A)-C(6)-H(6B)	109.5
C(4)-C(6)-H(6C)	109.5
H(6A)-C(6)-H(6C)	109.5
H(6B)-C(6)-H(6C)	109.5
C(9)-C(7)-C(8)	107.6(4)
C(9)-C(7)-P	113.9(3)
C(8)-C(7)-P	118.6(3)
C(9)-C(7)-H(7)	105.2
C(8)-C(7)-H(7)	105.2
P-C(7)-H(7)	105.2
C(7)-C(8)-H(8A)	109.5
C(7)-C(8)-H(8B)	109.5
H(8A)-C(8)-H(8B)	109.5
C(7)-C(8)-H(8C)	109.5
H(8A)-C(8)-H(8C)	109.5
H(8B)-C(8)-H(8C)	109.5
C(7)-C(9)-H(9A)	109.5
C(7)-C(9)-H(9B)	109.5
H(9A)-C(9)-H(9B)	109.5
C(7)-C(9)-H(9C)	109.5
H(9A)-C(9)-H(9C)	109.5
H(9B)-C(9)-H(9C)	109.5
C(15)-C(10)-C(11)	115.5(4)
C(15)-C(10)-Sn	125.6(3)
C(11)-C(10)-Sn	118.9(3)
C(12)-C(11)-C(10)	122.1(4)
C(12)-C(11)-H(11)	119.0
C(10)-C(11)-H(11)	119.0
C(13)-C(12)-C(11)	120.2(5)
C(13)-C(12)-H(12)	119.9
C(11)-C(12)-H(12)	119.9
C(14)-C(13)-C(12)	119.5(5)
C(14)-C(13)-H(13)	120.3
C(12)-C(13)-H(13)	120.2
C(13)-C(14)-C(15)	120.5(5)
C(13)-C(14)-H(14)	119.7
C(15)-C(14)-H(14)	119.7
C(14)-C(15)-C(10)	122.2(4)
C(14)-C(15)-H(15)	118.9
C(10)-C(15)-H(15)	118.9
C(17)-C(16)-C(21)	115.9(4)
C(17)-C(16)-Sn	121.8(3)
C(21)-C(16)-Sn	122.3(3)
C(16)-C(17)-C(18)	122.9(4)
C(16)-C(17)-H(17)	118.5
C(18)-C(17)-H(17)	118.5
C(19)-C(18)-C(17)	119.4(4)

C(19)-C(18)-H(18)	120.3
C(17)-C(18)-H(18)	120.3
C(20)-C(19)-C(18)	119.9(4)
C(20)-C(19)-H(19)	120.0
C(18)-C(19)-H(19)	120.0
C(19)-C(20)-C(21)	120.2(4)
C(19)-C(20)-H(20)	119.9
C(21)-C(20)-H(20)	119.9
C(20)-C(21)-C(16)	121.7(4)
C(20)-C(21)-H(21)	119.1
C(16)-C(21)-H(21)	119.1
C(27)-C(22)-C(23)	117.2(4)
C(27)-C(22)-Sn	122.2(3)
C(23)-C(22)-Sn	120.7(3)
C(24)-C(23)-C(22)	121.3(4)
C(24)-C(23)-H(23)	119.4
C(22)-C(23)-H(23)	119.4
C(25)-C(24)-C(23)	120.7(4)
C(25)-C(24)-H(24)	119.7
C(23)-C(24)-H(24)	119.7
C(24)-C(25)-C(26)	119.2(4)
C(24)-C(25)-H(25)	120.4
C(26)-C(25)-H(25)	120.4
C(27)-C(26)-C(25)	119.3(5)
C(27)-C(26)-H(26)	120.3
C(25)-C(26)-H(26)	120.3
C(22)-C(27)-C(26)	122.3(4)
C(22)-C(27)-H(27)	118.8
C(26)-C(27)-H(27)	118.8

Symmetry transformations used to generate equivalent atoms:
#1 -x+2,y,-z+1/2

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 2.
The anisotropic displacement factor exponent takes the form:
 $-2 \pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
Os	26(1)	20(1)	25(1)	0	8(1)	0
Sn	29(1)	24(1)	30(1)	0(1)	11(1)	-2(1)
P	30(1)	24(1)	28(1)	-1(1)	8(1)	-2(1)
C(1)	39(3)	34(2)	24(2)	-5(2)	7(2)	-1(2)
C(2)	60(4)	42(3)	32(3)	1(2)	12(2)	-9(3)
C(3)	47(3)	49(3)	34(3)	-10(2)	17(2)	-2(3)
C(4)	31(3)	22(2)	35(3)	-2(2)	7(2)	0(2)
C(5)	46(3)	32(2)	42(3)	-1(2)	8(2)	-8(2)
C(6)	53(3)	27(2)	39(3)	5(2)	14(2)	-7(2)
C(7)	34(3)	25(2)	39(3)	1(2)	6(2)	5(2)
C(8)	39(3)	54(3)	46(3)	-3(3)	0(2)	10(3)
C(9)	34(3)	42(3)	52(3)	-10(2)	11(2)	-5(2)
C(10)	27(2)	33(2)	32(2)	-7(2)	10(2)	-3(2)
C(11)	47(3)	36(2)	43(3)	-3(2)	20(3)	-3(2)
C(12)	41(3)	60(3)	36(3)	-4(2)	18(3)	-10(3)
C(13)	32(3)	75(4)	39(3)	-15(3)	13(2)	9(3)
C(14)	51(3)	46(3)	44(3)	-11(3)	6(3)	10(3)
C(15)	39(3)	33(2)	43(3)	-2(2)	6(2)	1(2)
C(16)	31(3)	26(2)	33(2)	3(2)	10(2)	-1(2)
C(17)	27(3)	35(2)	38(3)	2(2)	11(2)	-4(2)
C(18)	44(3)	38(3)	36(3)	-3(2)	9(2)	4(2)
C(19)	35(3)	36(2)	41(3)	-1(2)	1(2)	-5(2)
C(20)	26(3)	53(3)	48(3)	1(3)	7(2)	-8(2)
C(21)	37(3)	39(3)	37(3)	-4(2)	15(2)	-2(2)
C(22)	25(2)	28(2)	37(3)	3(2)	15(2)	-2(2)
C(23)	31(3)	37(2)	39(3)	2(2)	15(2)	-4(2)
C(24)	45(3)	27(2)	53(3)	-1(2)	18(3)	-3(2)
C(25)	61(4)	34(2)	47(3)	13(2)	11(3)	9(3)
C(26)	59(4)	44(3)	37(3)	8(2)	10(3)	3(3)
C(27)	49(3)	28(2)	44(3)	0(2)	17(2)	1(2)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 2.

	x	y	z	U(eq)
H(01)	9470(30)	9288(16)	2941(17)	24(10)
H(02)	10550(30)	8651(17)	2962(17)	34(11)
H(1)	11921	10080	4403	39
H(2A)	11360	9224	4930	67
H(2B)	11758	8881	4353	67
H(2C)	10470	8972	4257	67
H(3A)	9595	10052	4010	63
H(3B)	10271	10706	3938	63
H(3C)	10409	10396	4662	63
H(4)	10667	10846	2971	36
H(5A)	12925	10985	3713	62
H(5B)	11900	11131	3985	62
H(5C)	12113	11593	3414	62
H(6A)	11447	11229	2213	59
H(6B)	11317	10446	2064	59
H(6C)	12495	10749	2466	59
H(7)	12704	8936	3532	41
H(8A)	14360	9426	4206	74
H(8B)	13385	9488	4538	74
H(8C)	13730	10121	4181	74
H(9A)	13665	9959	2974	65
H(9B)	12810	9441	2512	65
H(9C)	13947	9179	3016	65
H(11)	7670	7638	3944	49
H(12)	6694	7922	4672	54
H(13)	6365	9032	4854	58
H(14)	6983	9854	4286	58
H(15)	7943	9581	3545	48
H(17)	8289	7518	1530	40
H(18)	6906	7004	696	48
H(19)	5120	6977	769	48
H(20)	4735	7458	1678	52
H(21)	6129	7950	2525	44
H(23)	9112	6746	2658	41
H(24)	10056	5827	3214	49
H(25)	10983	5876	4350	58
H(26)	10948	6868	4935	57
H(27)	10079	7801	4359	47

Computational Details

DFT optimizations were performed using the Gaussian98¹ series of programs within the B3LYP functional.² Effective core potentials were used to represent the innermost electrons of the osmium, tin and phosphorus atoms.³ The basis set for the osmium, tin and phosphorus atoms was that associated with the pseudopotential with a standard valence double- ζ LANL2DZ contraction.¹ A set of d polarization functions were added for the tin and the phosphorus atoms to supplement the standard basis set.⁴ Hydrogen atoms directly bonded to the osmium were described by using the 6-31G(d,p)⁵ basis set. A 6-31G⁵ basis set was used for all of the remaining hydrogen atoms. Calculations on the real system were carried out by using the IMOMM⁶ method, with a program built from modified versions of Gaussian98¹ for the quantum mechanics part and mm3(92)⁷ for the molecular mechanics part. The osmium atom and all of the atoms directly bonded to it were described at the quantum mechanics level, while phosphine and stannyl substituents have been treated at the molecular mechanics level. The QM part of the calculations was done at the B3LYP level using the same basis set as in the model complex. The MM part calculations used the mm3(92) force field⁸ with van der Waals parameters for the osmium taken from the UFF force field⁹. Torsional contributions involving dihedral angles with the metal center were set to zero. All of the geometrical parameters were optimized except the bond distances of atoms involved in the QM-MM frontier. The frozen values were 1.41 Å for the P-H bonds and 1.74 Å for the Sn-H bonds in the quantum part and the crystallographic values for the P-C and the Sn-C bonds in the MM part.

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