Supplemental Material

Single-Crystal X-ray Structure determination of [Pd(C3H5)(PPh2C6H4OH)]THF [SbF6]

Cationic methallylnickel and (meth)allylpalladium 2-phosphinophenol complexes: Synthesis, structural aspects and use in oligomerization of ethylene

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Table 1. Crystal data and structure refinement for [Pd(C3H5)(PPh2C6H4OH)]THF [SbF6].

Empirical formula	C25 H28 F6 O2 P Pd Sb	
Formula weight	733.59	
Temperature	213(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 8.7047(2) Å	α= 90°.
	b = 11.4662(2) Å	β= 90°.
	c = 27.3342(7) Å	γ = 90°.
Volume	2728.22(11) $Å^3$	
Z	4	
Density (calculated)	1.786 Mg/m^3	
Absorption coefficient	1.770 mm^{-1}	
F(000)	1440	
Crystal size	0.31 x 0.17 x 0.10 mm^3	
Theta range for data collection	3.03 to 32.85°.	
Index ranges	-13<=h<=12, -17<=k<=9, -40<=l<=41	
Reflections collected	43115	
Independent reflections	10100 [R(int) = 0.0312]	
Completeness to theta = 32.85°	99.6 %	
Absorption correction	SADABS	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	10100 / 0 / 326	
Goodness-of-fit on F^2	1.054	
Final R indices [I>2sigma(I)]	R1 = 0.0315, wR2 = 0.0723	
R indices (all data)	R1 = 0.0475, wR2 = 0.0778	
Absolute structure parameter	0.00(3)	
Largest diff. peak and hole	0.707 and -0.425 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for [Pd(C3H5)(PPh2C6H4OH)]THF [SbF6]. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Sb(1)	2264(1)	3684(1)	-1457(1)	50(1)
F(1)	3264(3)	2479(2)	-1787(1)	89(1)
F(2)	674(3)	3636(3)	-1910(1)	94(1)
F(3)	1270(3)	4857(2)	-1111(1)	88(1)
F(4)	3244(3)	4759(3)	-1868(1)	105(1)
F(5)	3902(4)	3745(3)	-1028(1)	119(1)
F(6)	1247(5)	2580(3)	-1090(1)	125(1)
Pd(1)	3038(1)	4567(1)	1440(1)	34(1)
P(1)	4361(1)	6263(1)	1318(1)	31(1)
C(1)	3648(4)	4067(3)	2136(1)	58(1)
C(2)	2225(4)	3506(3)	2027(1)	60(1)
C(3)	2111(4)	2830(3)	1640(1)	59(1)
C(4)	4354(3)	6332(2)	655(1)	35(1)
C(5)	3368(3)	5582(3)	408(1)	38(1)
O(1)	2510(3)	4808(2)	674(1)	53(1)
C(6)	3277(3)	5609(3)	-103(1)	48(1)
C(7)	4170(4)	6372(3)	-360(1)	52(1)
C(8)	5190(4)	7104(3)	-121(1)	54(1)
C(9)	5268(3)	7093(3)	381(1)	47(1)
C(10)	3473(3)	7604(2)	1528(1)	33(1)
C(11)	2850(4)	8423(3)	1212(1)	46(1)
C(12)	2113(4)	9410(3)	1395(1)	54(1)
C(13)	2034(3)	9579(3)	1892(1)	51(1)
C(14)	2624(4)	8778(3)	2204(1)	53(1)
C(15)	3336(3)	7775(3)	2026(1)	45(1)
C(16)	6361(3)	6345(2)	1494(1)	35(1)
C(17)	7079(3)	7402(3)	1607(1)	44(1)
C(18)	8637(4)	7401(3)	1734(1)	55(1)
C(19)	9445(3)	6371(4)	1743(1)	66(1)
C(20)	8738(3)	5338(3)	1630(2)	67(1)

C(21)	7184(3)	5319(3)	1512(1)	50(1)
O(2)	27(3)	4181(3)	287(1)	79(1)
C(22)	-271(6)	3156(5)	12(2)	102(2)
C(23)	-1699(6)	3398(7)	-265(2)	128(3)
C(24)	-2483(6)	4300(4)	24(2)	105(2)
C(25)	-1247(7)	4917(6)	222(3)	147(3)

Table 3. Bond lengths [Å] and angles [°] for [Pd(C3H5)(PPh2C6H4OH)]THF [SbF6].

Sb(1)-F(6)	1.843(3)
Sb(1)-F(5)	1.848(2)
Sb(1)-F(3)	1.858(2)
Sb(1)-F(2)	1.858(2)
Sb(1)-F(1)	1.866(2)
Sb(1)-F(4)	1.873(3)
Pd(1)-C(1)	2.056(3)
Pd(1)-C(2)	2.134(3)
Pd(1)-O(1)	2.1606(18)
Pd(1)-C(3)	2.217(3)
Pd(1)-P(1)	2.2849(7)
P(1)-C(16)	1.809(2)
P(1)-C(4)	1.812(2)
P(1)-C(10)	1.814(3)
C(1)-C(2)	1.427(5)
C(2)-C(3)	1.316(5)
C(4)-C(5)	1.390(4)
C(4)-C(9)	1.399(4)
C(5)-O(1)	1.369(3)
C(5)-C(6)	1.400(4)
C(6)-C(7)	1.365(5)
C(7)-C(8)	1.385(5)
C(8)-C(9)	1.374(4)
C(10)-C(11)	1.386(4)
C(10)-C(15)	1.381(4)
C(11)-C(12)	1.395(4)
C(12)-C(13)	1.372(4)
C(13)-C(14)	1.355(5)
C(14)-C(15)	1.394(4)
C(16)-C(21)	1.378(4)
C(16)-C(17)	1.398(4)
C(17)-C(18)	1.400(4)
C(18)-C(19)	1.375(5)
C(19)-C(20)	1.370(5)

C(20)-C(21)	1.391(4)
O(2)-C(25)	1.405(6)
O(2)-C(22)	1.419(6)
C(22)-C(23)	1.482(7)
C(23)-C(24)	1.470(7)
C(24)-C(25)	1.396(7)

F(6)-Sb(1)-F(5)	92.93(19)
F(6)-Sb(1)-F(3)	89.80(13)
F(5)-Sb(1)-F(3)	90.51(12)
F(6)-Sb(1)-F(2)	89.09(18)
F(5)-Sb(1)-F(2)	177.62(14)
F(3)-Sb(1)-F(2)	90.75(12)
F(6)-Sb(1)-F(1)	88.79(15)
F(5)-Sb(1)-F(1)	88.62(13)
F(3)-Sb(1)-F(1)	178.31(13)
F(2)-Sb(1)-F(1)	90.17(12)
F(6)-Sb(1)-F(4)	176.13(16)
F(5)-Sb(1)-F(4)	90.27(16)
F(3)-Sb(1)-F(4)	92.36(15)
F(2)-Sb(1)-F(4)	87.67(14)
F(1)-Sb(1)-F(4)	89.10(13)
C(1)-Pd(1)-C(2)	39.77(14)
C(1)-Pd(1)-O(1)	170.64(13)
C(2)-Pd(1)-O(1)	136.93(12)
C(1)-Pd(1)-C(3)	67.40(14)
C(2)-Pd(1)-C(3)	35.13(14)
O(1)-Pd(1)-C(3)	106.03(11)
C(1)-Pd(1)-P(1)	104.03(11)
C(2)-Pd(1)-P(1)	139.66(12)
O(1)-Pd(1)-P(1)	81.76(6)
C(3)-Pd(1)-P(1)	169.77(9)
C(16)-P(1)-C(4)	105.50(11)
C(16)-P(1)-C(10)	106.37(12)
C(4)-P(1)-C(10)	106.13(12)
C(16)-P(1)-Pd(1)	119.34(9)

C(4)-P(1)-Pd(1)	100.43(9)
C(10)-P(1)-Pd(1)	117.44(8)
C(2)-C(1)-Pd(1)	73.06(18)
C(3)-C(2)-C(1)	119.9(3)
C(3)-C(2)-Pd(1)	75.9(2)
C(1)-C(2)-Pd(1)	67.17(18)
C(2)-C(3)-Pd(1)	69.0(2)
C(5)-C(4)-C(9)	118.4(2)
C(5)-C(4)-P(1)	117.44(19)
C(9)-C(4)-P(1)	124.2(2)
O(1)-C(5)-C(4)	118.7(2)
O(1)-C(5)-C(6)	120.9(3)
C(4)-C(5)-C(6)	120.4(3)
C(5)-O(1)-Pd(1)	118.75(15)
C(7)-C(6)-C(5)	119.7(3)
C(6)-C(7)-C(8)	120.8(3)
C(9)-C(8)-C(7)	119.7(3)
C(8)-C(9)-C(4)	120.9(3)
C(11)-C(10)-C(15)	119.0(3)
C(11)-C(10)-P(1)	122.9(2)
C(15)-C(10)-P(1)	117.9(2)
C(10)-C(11)-C(12)	120.3(3)
C(13)-C(12)-C(11)	119.6(3)
C(14)-C(13)-C(12)	120.5(3)
C(13)-C(14)-C(15)	120.6(3)
C(10)-C(15)-C(14)	119.9(3)
C(21)-C(16)-C(17)	120.0(2)
C(21)-C(16)-P(1)	117.72(19)
C(17)-C(16)-P(1)	122.3(2)
C(16)-C(17)-C(18)	119.2(3)
C(19)-C(18)-C(17)	120.0(3)
C(20)-C(19)-C(18)	120.6(3)
C(19)-C(20)-C(21)	120.2(3)
C(16)-C(21)-C(20)	120.0(3)
C(25)-O(2)-C(22)	106.6(4)
O(2)-C(22)-C(23)	105.6(5)

C(22)-C(23)-C(24)	104.2(4)
C(25)-C(24)-C(23)	102.0(5)
C(24)-C(25)-O(2)	110.7(5)

Table 4. Anisotropic displacement parameters ($Å^2$x 10^3)for [Pd(C3H5)(PPh2C6H4OH)]THF [SbF6]. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

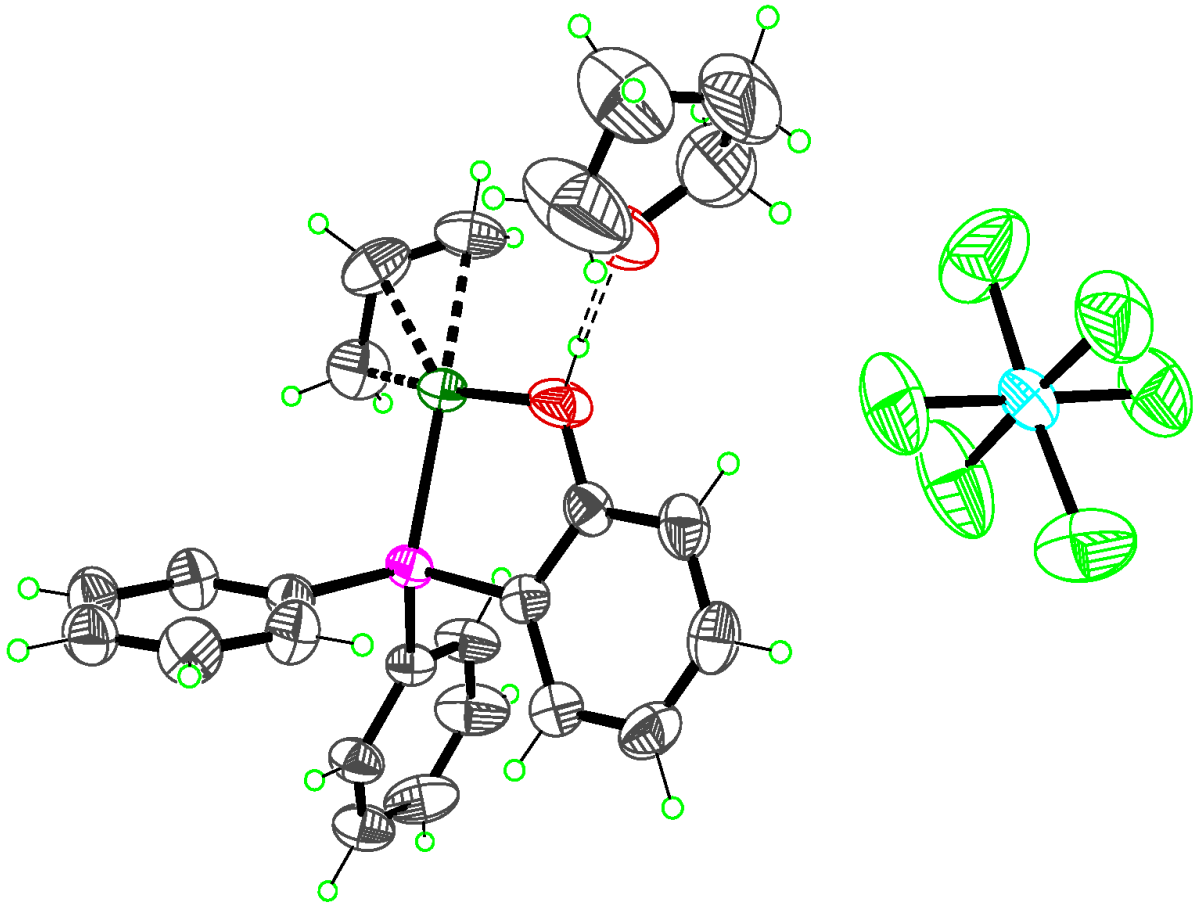
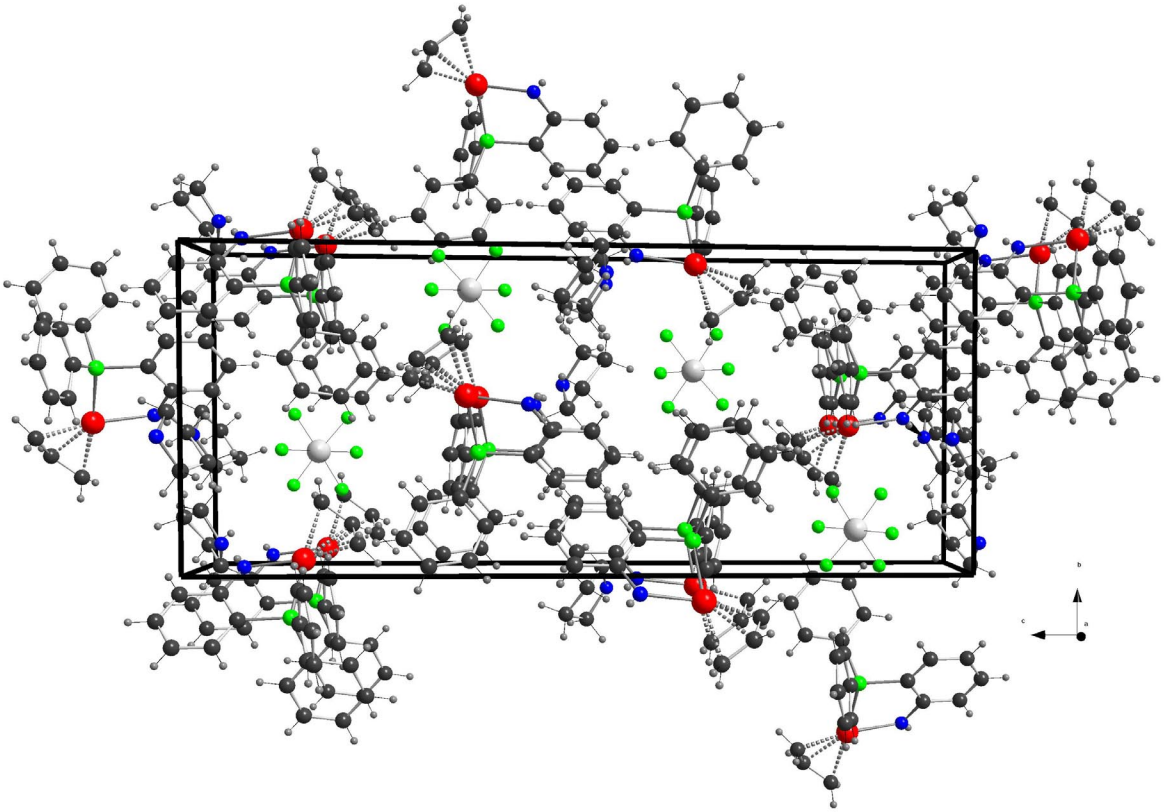
	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Sb(1)	55(1)	39(1)	56(1)	-7(1)	-9(1)	-1(1)
F(1)	95(2)	65(2)	107(2)	-30(1)	-6(2)	18(1)
F(2)	64(1)	133(2)	84(1)	-26(2)	-23(1)	1(2)
F(3)	98(2)	76(2)	91(2)	-35(1)	-19(1)	30(2)
F(4)	95(2)	84(2)	136(2)	23(2)	17(2)	-16(2)
F(5)	118(2)	124(3)	116(2)	-52(2)	-70(2)	47(2)
F(6)	175(3)	81(2)	120(2)	28(2)	63(2)	-1(2)
Pd(1)	31(1)	32(1)	39(1)	4(1)	-4(1)	-3(1)
P(1)	30(1)	30(1)	34(1)	2(1)	-4(1)	-3(1)
C(1)	65(2)	61(2)	46(2)	17(2)	-14(2)	-2(2)
C(2)	54(2)	64(2)	63(2)	33(2)	10(2)	5(2)
C(3)	57(2)	40(2)	79(2)	19(2)	-8(2)	-15(2)
C(4)	33(1)	35(1)	38(1)	2(1)	-1(1)	1(1)
C(5)	44(1)	37(2)	35(1)	0(1)	-6(1)	0(1)
O(1)	54(1)	64(2)	43(1)	6(1)	-16(1)	-27(1)
C(6)	52(2)	57(2)	36(1)	-6(1)	-8(1)	2(1)
C(7)	62(2)	61(2)	34(1)	2(1)	-2(1)	11(2)
C(8)	56(2)	61(2)	44(2)	14(2)	8(1)	-7(2)
C(9)	48(2)	46(2)	46(2)	-1(1)	4(1)	-4(1)
C(10)	29(1)	34(1)	38(1)	0(1)	-2(1)	-3(1)
C(11)	51(2)	47(2)	40(1)	4(1)	1(1)	9(1)
C(12)	58(2)	41(2)	63(2)	11(2)	2(2)	14(1)
C(13)	47(1)	41(2)	65(2)	-7(2)	5(1)	4(2)
C(14)	57(2)	56(2)	47(1)	-12(2)	-1(1)	-1(2)
C(15)	51(2)	45(2)	40(1)	-1(1)	-6(1)	7(1)
C(16)	30(1)	35(1)	40(1)	5(1)	-3(1)	-4(1)
C(17)	34(1)	40(2)	57(2)	-2(1)	-5(1)	-7(1)
C(18)	39(2)	56(2)	69(2)	0(2)	-8(1)	-15(1)
C(19)	26(1)	83(3)	90(2)	23(2)	-7(1)	-10(2)
C(20)	34(1)	52(2)	114(3)	19(2)	-7(2)	5(2)
C(21)	34(1)	34(1)	84(2)	10(2)	-6(1)	-2(1)

O(2)	60(2)	95(2)	81(2)	-16(2)	-20(1)	-20(1)
C(22)	78(3)	108(4)	120(4)	-25(4)	4(3)	5(3)
C(23)	66(3)	186(7)	131(5)	-74(5)	-26(3)	-20(4)
C(24)	83(3)	86(4)	145(5)	-19(4)	-41(4)	19(3)
C(25)	80(4)	107(5)	255(9)	-69(5)	-33(5)	5(3)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for [Pd(C3H5)(PPh2C6H4OH)]THF [SbF6].

	x	y	z	U(eq)
H(1B)	4670	3767	2177	69
H(1C)	3684	4653	2386	69
H(2A)	1285	3737	2206	72
H(3A)	1071	2647	1526	70
H(3B)	2824	2170	1620	70
H(1A)	1659	4585	553	64
H(6A)	2603	5104	-268	58
H(7A)	4093	6400	-703	62
H(8A)	5826	7607	-302	64
H(9A)	5945	7603	542	56
H(11A)	2924	8312	872	55
H(12A)	1674	9957	1181	65
H(13A)	1567	10256	2016	61
H(14A)	2552	8900	2543	64
H(15A)	3723	7215	2245	54
H(17A)	6523	8104	1597	53
H(18A)	9132	8105	1813	66
H(19A)	10491	6375	1828	79
H(20A)	9304	4640	1633	80
H(21A)	6695	4607	1444	60
H(22A)	-413	2485	230	123
H(22B)	581	2990	-211	123
H(23A)	-2334	2695	-292	154
H(23B)	-1463	3683	-595	154
H(24A)	-3121	4801	-184	126
H(24B)	-3122	3955	281	126
H(25B)	-1548	5251	538	177
H(25A)	-970	5560	3	177

Molecular structure of **4a·THF** (with anion).



Package of **4a·THF** in the crystal.