Reductive Elimination from Metal Phosphonate Complexes: Circumvention of Competing Protonolysis Reactions

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Supplemental Information

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Crystallographic Experimental Section for Compound 5.

Data Collection

A colorless air-sensitive crystal with approximate dimensions 0.39 x 0.30 x 0.19 mm^3 was selected under oil under ambient conditions and attached to the tip of a nylon loop. The crystal was mounted in a stream of cold nitrogen at 100(2) K and centered in the X-ray beam by using a video camera.

The crystal evaluation and data collection were performed on a Bruker CCD-1000 diffractometer with Mo K_α (λ = 0.71073 Å) radiation and the diffractometer to crystal distance of 4.9 cm.

The initial cell constants were obtained from three series of ω scans at different starting angles. Each series consisted of 20 frames collected at intervals of 0.3º in a 6º range about ω with the exposure time of 8 seconds per frame. A total of 104 reflections was obtained. The reflections were successfully indexed by an automated indexing routine built in the SMART program. The final cell constants were calculated from a set of 27449 strong reflections from the actual data collection.

The data were collected by using the hemisphere data collection routine. The reciprocal space was surveyed to the extent of a full sphere to a resolution of 0.80 Å. A total of 42244 data were harvested by collecting nine sets of frames with 0.3º scans in ω and φ with an exposure time 30 sec per frame. These highly redundant datasets were corrected for Lorentz and polarization effects. The absorption correction was based on fitting a function to the empirical transmission surface as sampled by multiple equivalent measurements.[1]

Structure Solution and Refinement

The systematic absences in the diffraction data were consistent for the space groups $P\bar{1}$ and $P1$. The *E*-statistics strongly suggested the non-centrosymmetric space group $P1$ that yielded chemically reasonable and computationally stable results of refinement.[1] A successful solution by the direct methods provided most non-hydrogen atoms from the *E*-map. The remaining non-hydrogen atoms were located in an alternating series of least-squares cycles and difference Fourier maps. All non-hydrogen atoms were refined with anisotropic displacement

coefficients. All hydrogen atoms were included in the structure factor calculation at idealized positions and were allowed to ride on the neighboring atoms with relative isotropic displacement coefficients. There are two symmetry independent Pd complexes and two symmetry independent solvate molecules of dichloromethane in the unit cell. The compositions of the Pd complexes are identical but the geometries differ most noticeable in the position of one of the Ph rings (Figure 4).

The final least-squares refinement of 885 parameters against 15070 data resulted in residuals R (based on F^2 for $I \geq 2\sigma$) and wR (based on F^2 for all data) of 0.0272 and 0.0659, respectively. The final difference Fourier map was featureless.

The ORTEP diagrams are drawn with 50% probability ellipsoids.

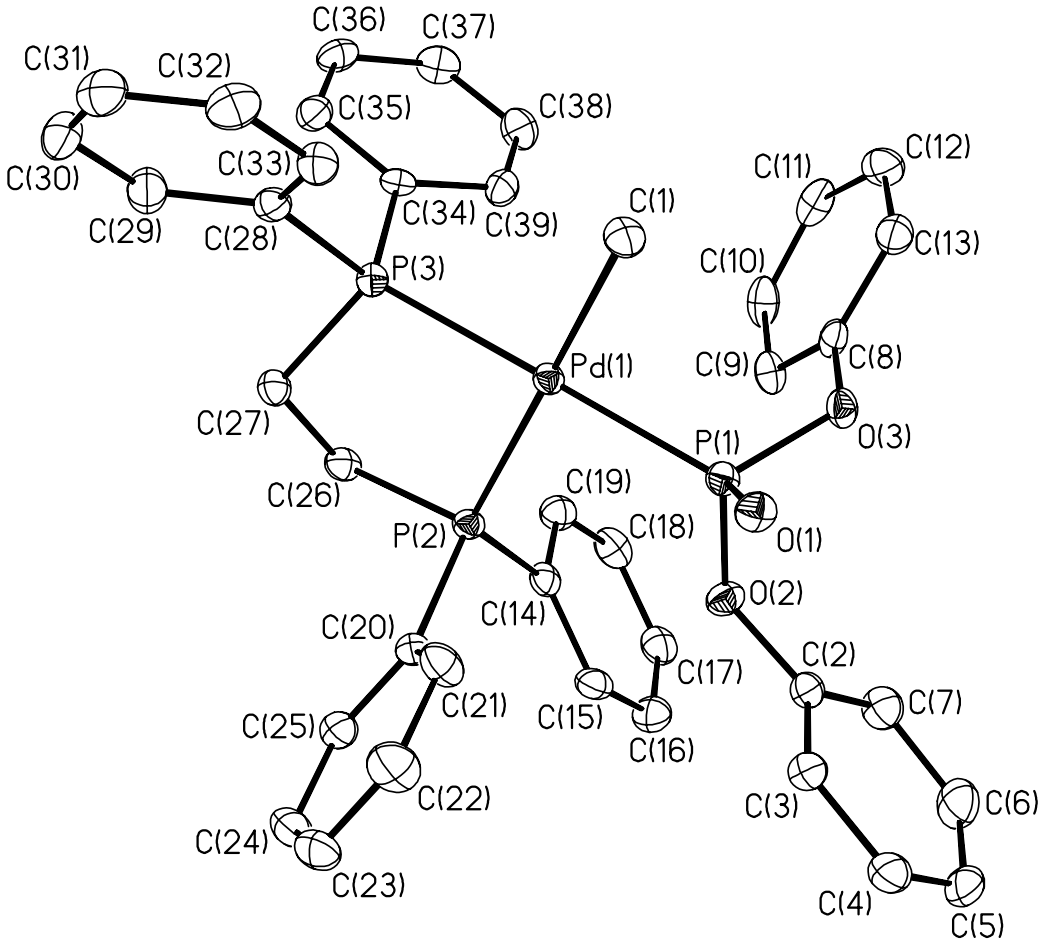

Figure 1. The first molecule of **5**.

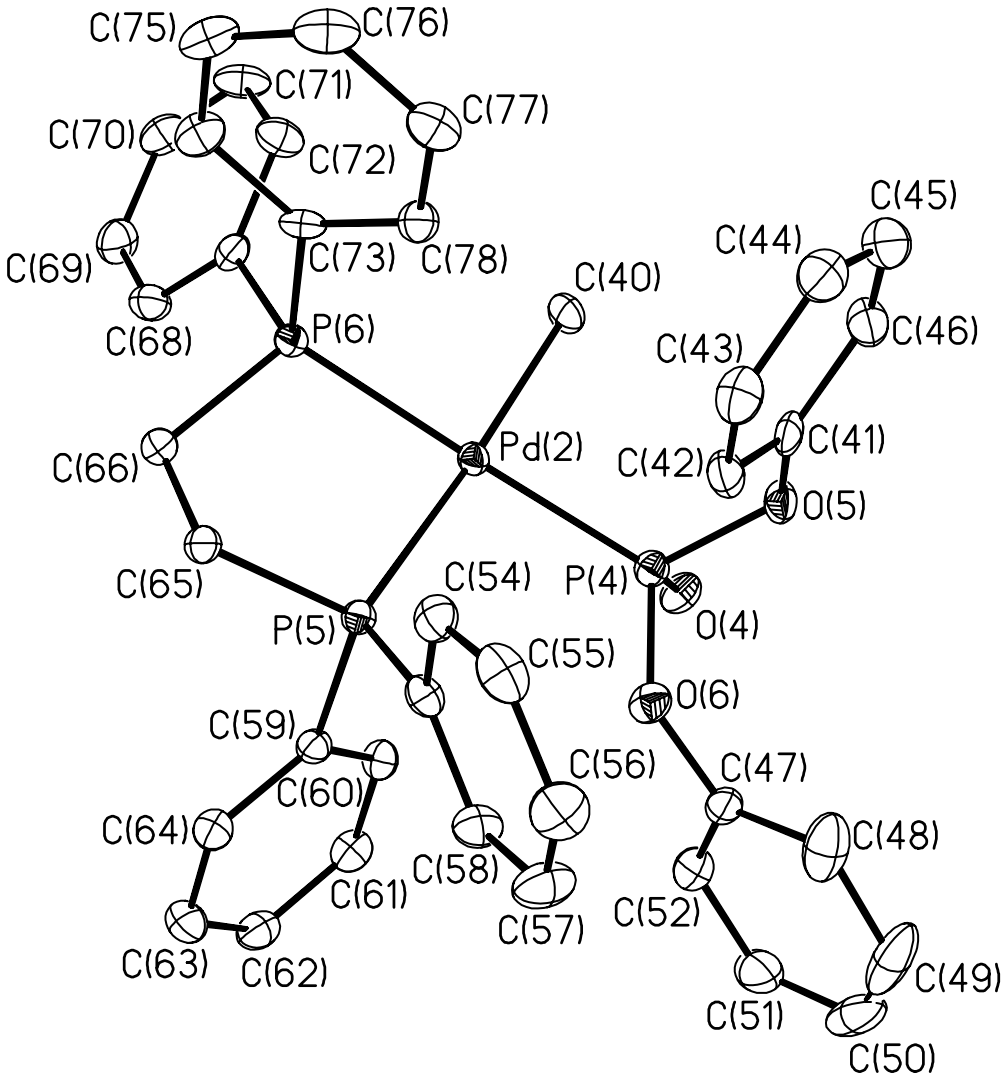

Figure 2. The second molecule of the Pd complex.

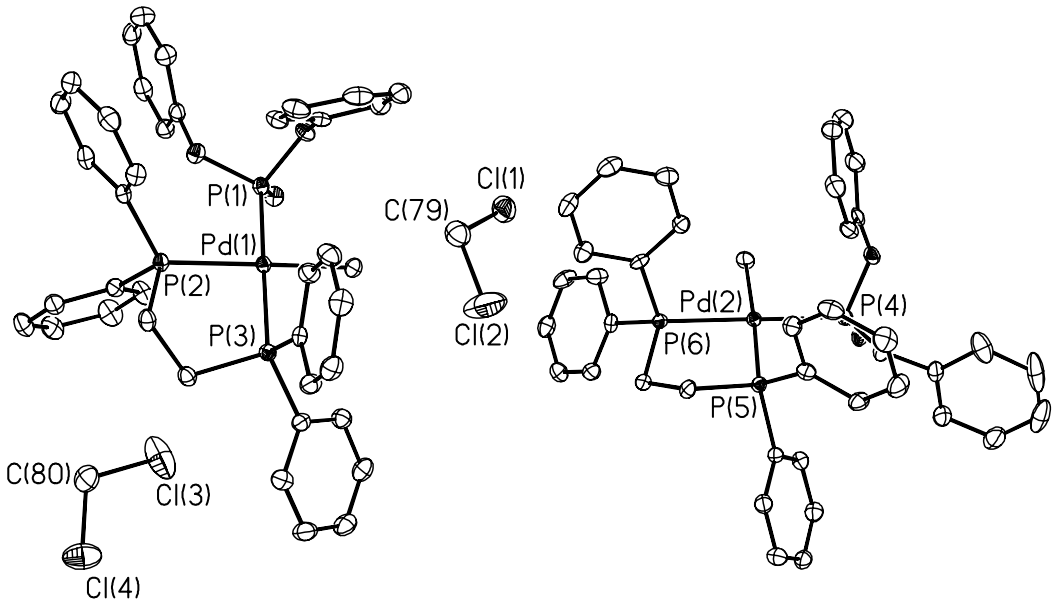

Figure 3. The composition of the unit cell.

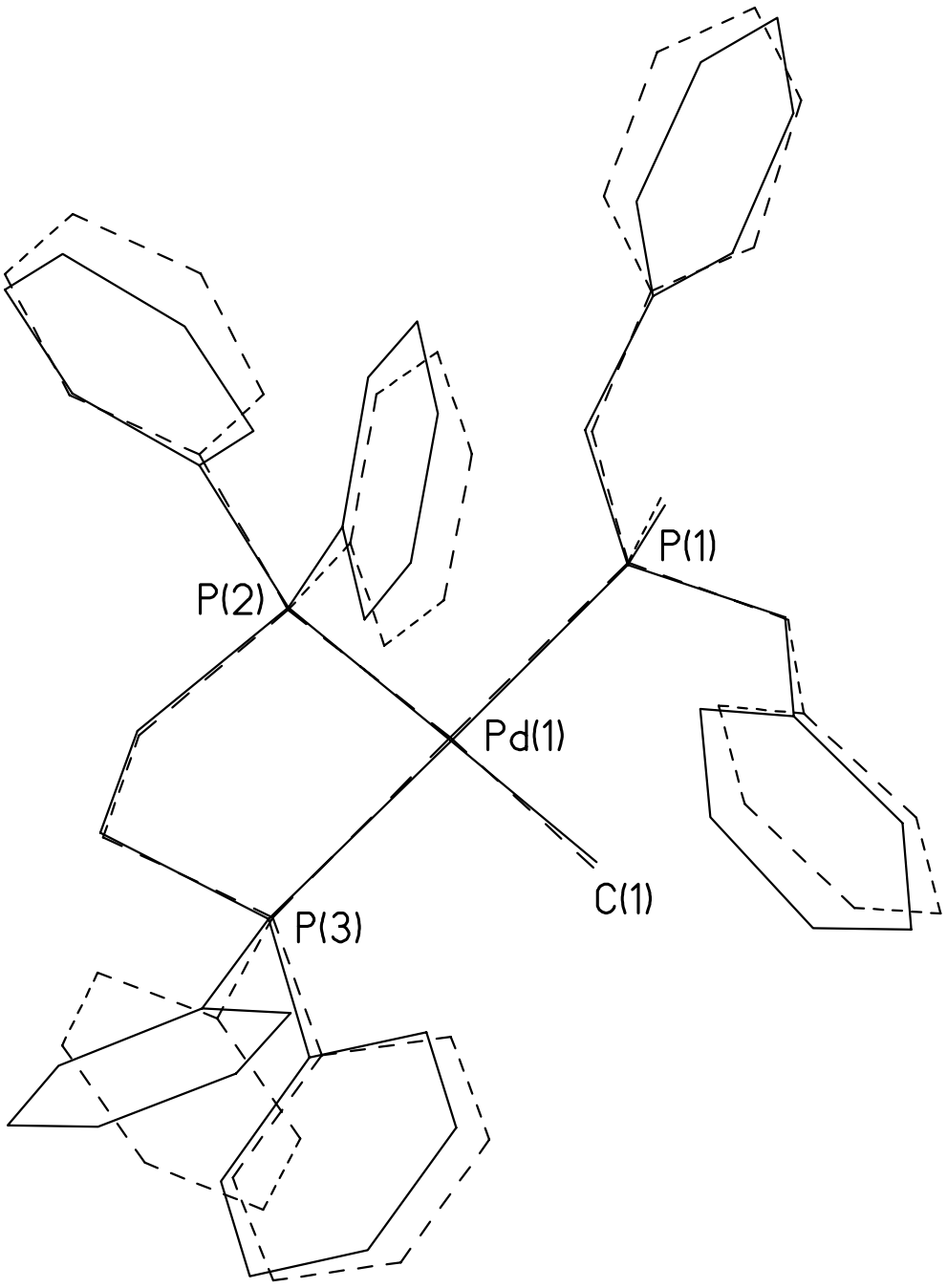

Figure 4. The two molecules of the Pd complexes superimposed.

Table 1. Crystal data and structure refinement for compound **5**.

Identification code	sto06	
Empirical formula	$C_{40}\ H_{39}\ Cl_2\ O_3\ P_3\ Pd$	
Formula weight	837.92	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P1	
Unit cell dimensions	a = 9.4090(8) Å	α= 93.0130(10)°.
	b = 11.6613(10) Å	β= 104.8470(10)°.
	c = 17.6423(15) Å	γ = 96.9180(10)°.
Volume	1850.4(3) $Å^3$	
Z	2	
Density (calculated)	1.504 Mg/m^3	
Absorption coefficient	0.814 mm^{-1}	
F(000)	856	
Crystal size	0.39 x 0.30 x 0.19 mm^3	
Theta range for data collection	2.05 to 26.50°.	
Index ranges	-11<=h<=11, -14<=k<=14, -22<=l<=22	
Reflections collected	42244	
Independent reflections	15070 [R(int) = 0.0353]	
Completeness to theta = 26.50°	99.0 %	
Absorption correction	Multiscan with SADABS	
Max. and min. transmission	0.8607 and 0.7420	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	15070 / 3 / 885	
Goodness-of-fit on F^2	1.030	
Final R indices [I>2sigma(I)]	R1 = 0.0272, wR2 = 0.0653	
R indices (all data)	R1 = 0.0283, wR2 = 0.0659	
Absolute structure parameter	-0.019(10)	
Largest diff. peak and hole	1.019 and -0.439 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for compound **5**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Pd(1)	3810(1)	1565(1)	4983(1)	13(1)
Pd(2)	4970(1)	8854(1)	74(1)	13(1)
Cl(1)	865(1)	3695(1)	2429(1)	35(1)
Cl(2)	181(2)	4855(1)	3766(1)	68(1)
Cl(3)	7755(1)	3189(1)	7533(1)	64(1)
Cl(4)	8191(1)	3714(1)	9212(1)	53(1)
P(1)	4340(1)	285(1)	4102(1)	14(1)
P(2)	3811(1)	325(1)	5977(1)	14(1)
P(3)	3435(1)	2952(1)	5890(1)	14(1)
P(4)	4442(1)	10075(1)	-902(1)	15(1)
P(5)	4053(1)	9812(1)	1000(1)	14(1)
P(6)	5656(1)	7610(1)	1041(1)	14(1)
O(1)	5732(2)	534(2)	3857(1)	18(1)
O(2)	4237(2)	-1008(2)	4437(1)	18(1)
O(3)	3010(2)	33(2)	3267(1)	17(1)
O(4)	5681(2)	10728(2)	-1147(1)	21(1)
O(5)	3329(2)	9410(2)	-1731(1)	19(1)
O(6)	3394(2)	10988(2)	-667(1)	19(1)
C(1)	3857(3)	2709(2)	4097(2)	20(1)
C(2)	4725(3)	-1946(2)	4120(2)	19(1)
C(3)	6230(3)	-1961(3)	4194(2)	20(1)
C(4)	6710(3)	-2949(3)	3916(2)	26(1)
C(5)	5687(4)	-3914(3)	3578(2)	29(1)
C(6)	4200(4)	-3893(3)	3512(2)	31(1)
C(7)	3697(3)	-2915(3)	3777(2)	25(1)
C(8)	1535(3)	109(2)	3232(2)	17(1)
C(9)	876(3)	-339(3)	3790(2)	20(1)
C(10)	-586(3)	-184(3)	3741(2)	25(1)
C(11)	-1395(3)	374(3)	3146(2)	25(1)
C(12)	-740(3)	769(3)	2567(2)	25(1)
C(13)	731(3)	634(2)	2607(2)	21(1)
C(14)	2630(3)	-1074(2)	5803(2)	17(1)
C(15)	3183(3)	-2116(2)	5728(2)	20(1)
C(16)	2233(3)	-3153(3)	5548(2)	25(1)
C(17)	715(3)	-3164(3)	5457(2)	24(1)
C(18)	155(3)	-2133(3)	5532(2)	25(1)
C(19)	1096(3)	-1098(3)	5695(2)	21(1)
C(20)	5678(3)	71(2)	6462(2)	17(1)
C(21)	6825(3)	411(3)	6117(2)	23(1)
C(22)	8281(3)	266(3)	6489(2)	30(1)
C(23)	8593(3)	-222(3)	7199(2)	27(1)
C(24)	7476(3)	-545(3)	7553(2)	26(1)
C(25)	6018(3)	-406(2)	7189(2)	21(1)
C(26)	3174(3)	1072(2)	6755(2)	18(1)
C(27)	3801(3)	2348(2)	6855(2)	18(1)
C(28)	4541(3)	4387(2)	6131(2)	17(1)
C(29)	4751(3)	5001(3)	6868(2)	25(1)
C(30)	5671(3)	6058(3)	7061(2)	26(1)
C(31)	6413(3)	6503(3)	6526(2)	27(1)
C(32)	6205(3)	5909(3)	5809(2)	25(1)
C(33)	5277(3)	4852(2)	5606(2)	21(1)

C(34)	1470(3)	3115(2)	5680(2)	15(1)
C(35)	921(3)	3917(2)	6111(2)	20(1)
C(36)	-602(3)	3929(3)	5968(2)	23(1)
C(37)	-1594(3)	3139(3)	5398(2)	24(1)
C(38)	-1069(3)	2338(3)	4967(2)	24(1)
C(39)	458(3)	2332(2)	5108(2)	20(1)
C(40)	5738(3)	7870(3)	-739(2)	19(1)
C(41)	2201(3)	8514(2)	-1759(2)	18(1)
C(42)	1298(3)	8506(2)	-1250(2)	21(1)
C(43)	173(3)	7583(3)	-1322(2)	26(1)
C(44)	-65(3)	6683(3)	-1910(2)	27(1)
C(45)	826(4)	6719(3)	-2426(2)	30(1)
C(46)	1962(3)	7621(3)	-2352(2)	25(1)
C(47)	3218(3)	12040(2)	-1003(2)	20(1)
C(48)	1864(4)	12168(3)	-1499(2)	36(1)
C(49)	1674(4)	13270(4)	-1777(2)	47(1)
C(50)	2781(5)	14154(3)	-1579(2)	43(1)
C(51)	4132(5)	14026(3)	-1095(2)	38(1)
C(52)	4350(4)	12962(3)	-803(2)	28(1)
C(53)	2112(3)	10058(2)	741(2)	17(1)
C(54)	1008(3)	9103(2)	616(2)	20(1)
C(55)	-481(3)	9230(3)	355(2)	25(1)
C(56)	-888(3)	10312(3)	206(2)	28(1)
C(57)	196(3)	11266(3)	334(2)	29(1)
C(58)	1691(3)	11151(2)	599(2)	21(1)
C(59)	5133(3)	11207(2)	1424(2)	15(1)
C(60)	6074(3)	11777(2)	1020(2)	17(1)
C(61)	6879(3)	12849(2)	1318(2)	21(1)
C(62)	6794(3)	13366(3)	2024(2)	22(1)
C(63)	5894(3)	12799(3)	2442(2)	24(1)
C(64)	5054(3)	11727(2)	2143(2)	20(1)
C(65)	4145(3)	8901(2)	1830(2)	17(1)
C(66)	5545(3)	8303(2)	1975(2)	18(1)
C(67)	7442(3)	7069(2)	1276(2)	17(1)
C(68)	8636(3)	7635(3)	1867(2)	22(1)
C(69)	10018(3)	7259(3)	2007(2)	26(1)
C(70)	10235(3)	6337(3)	1555(2)	24(1)
C(71)	9054(3)	5761(3)	968(2)	26(1)
C(72)	7662(3)	6121(3)	820(2)	23(1)
C(73)	4278(3)	6327(2)	848(2)	16(1)
C(74)	4397(3)	5425(2)	1341(2)	23(1)
C(75)	3316(3)	4466(3)	1181(2)	26(1)
C(76)	2098(3)	4392(3)	538(2)	26(1)
C(77)	1955(3)	5285(3)	44(2)	23(1)
C(78)	3046(3)	6250(2)	198(2)	18(1)
C(79)	242(4)	3503(3)	3285(2)	32(1)
C(80)	7826(4)	2602(3)	8443(2)	35(1)

Table 3. Bond lengths [Å] and angles [°] for compound **5**.

Pd(1)-C(1)	2.114(3)	C(21)-C(22)	1.393(4)
Pd(1)-P(1)	2.2871(7)	C(22)-C(23)	1.381(5)
Pd(1)-P(2)	2.3301(8)	C(23)-C(24)	1.379(4)
Pd(1)-P(3)	2.3352(7)	C(24)-C(25)	1.390(4)
Pd(2)-C(40)	2.110(3)	C(26)-C(27)	1.516(4)
Pd(2)-P(4)	2.2869(8)	C(28)-C(33)	1.386(4)
Pd(2)-P(6)	2.3128(7)	C(28)-C(29)	1.406(4)
Pd(2)-P(5)	2.3293(7)	C(29)-C(30)	1.390(4)
Cl(1)-C(79)	1.770(3)	C(30)-C(31)	1.396(4)
Cl(2)-C(79)	1.766(3)	C(31)-C(32)	1.366(4)
Cl(3)-C(80)	1.767(4)	C(32)-C(33)	1.394(4)
Cl(4)-C(80)	1.762(4)	C(34)-C(35)	1.400(4)
P(1)-O(1)	1.4827(19)	C(34)-C(39)	1.401(4)
P(1)-O(2)	1.650(2)	C(35)-C(36)	1.392(4)
P(1)-O(3)	1.6553(19)	C(36)-C(37)	1.395(4)
P(2)-C(20)	1.814(3)	C(37)-C(38)	1.387(4)
P(2)-C(14)	1.824(3)	C(38)-C(39)	1.394(4)
P(2)-C(26)	1.850(3)	C(41)-C(42)	1.385(4)
P(3)-C(34)	1.826(3)	C(41)-C(46)	1.392(4)
P(3)-C(28)	1.829(3)	C(42)-C(43)	1.392(4)
P(3)-C(27)	1.844(3)	C(43)-C(44)	1.392(4)
P(4)-O(4)	1.484(2)	C(44)-C(45)	1.385(5)
P(4)-O(6)	1.642(2)	C(45)-C(46)	1.382(4)
P(4)-O(5)	1.6537(19)	C(47)-C(48)	1.378(4)
P(5)-C(59)	1.823(3)	C(47)-C(52)	1.381(4)
P(5)-C(53)	1.827(3)	C(48)-C(49)	1.414(5)
P(5)-C(65)	1.844(3)	C(49)-C(50)	1.338(6)
P(6)-C(73)	1.814(3)	C(50)-C(51)	1.367(6)
P(6)-C(67)	1.822(3)	C(51)-C(52)	1.386(4)
P(6)-C(66)	1.831(3)	C(53)-C(54)	1.395(4)
O(2)-C(2)	1.382(3)	C(53)-C(58)	1.395(4)
O(3)-C(8)	1.387(3)	C(54)-C(55)	1.387(4)
O(5)-C(41)	1.387(3)	C(55)-C(56)	1.381(4)
O(6)-C(47)	1.400(3)	C(56)-C(57)	1.383(4)
C(2)-C(3)	1.390(4)	C(57)-C(58)	1.390(4)
C(2)-C(7)	1.394(4)	C(59)-C(64)	1.400(4)
C(3)-C(4)	1.397(4)	C(59)-C(60)	1.402(4)
C(4)-C(5)	1.386(4)	C(60)-C(61)	1.383(4)
C(5)-C(6)	1.377(5)	C(61)-C(62)	1.379(4)
C(6)-C(7)	1.390(4)	C(62)-C(63)	1.391(4)
C(8)-C(9)	1.384(4)	C(63)-C(64)	1.395(4)
C(8)-C(13)	1.385(4)	C(65)-C(66)	1.533(4)
C(9)-C(10)	1.391(4)	C(67)-C(68)	1.390(4)
C(10)-C(11)	1.373(5)	C(67)-C(72)	1.400(4)
C(11)-C(12)	1.392(4)	C(68)-C(69)	1.390(4)
C(12)-C(13)	1.396(4)	C(69)-C(70)	1.372(4)
C(14)-C(15)	1.391(4)	C(70)-C(71)	1.383(4)
C(14)-C(19)	1.403(4)	C(71)-C(72)	1.389(4)
C(15)-C(16)	1.385(4)	C(73)-C(78)	1.396(4)
C(16)-C(17)	1.394(4)	C(73)-C(74)	1.396(4)
C(17)-C(18)	1.383(4)	C(74)-C(75)	1.383(4)
C(18)-C(19)	1.378(4)	C(75)-C(76)	1.381(4)
C(20)-C(21)	1.397(4)	C(76)-C(77)	1.390(4)
C(20)-C(25)	1.401(4)	C(77)-C(78)	1.393(4)

C(1)-Pd(1)-P(1)	82.90(8)	C(2)-C(3)-C(4)	119.7(3)
C(1)-Pd(1)-P(2)	178.72(9)	C(5)-C(4)-C(3)	119.9(3)
P(1)-Pd(1)-P(2)	97.38(3)	C(6)-C(5)-C(4)	120.0(3)
C(1)-Pd(1)-P(3)	95.25(8)	C(5)-C(6)-C(7)	121.1(3)
P(1)-Pd(1)-P(3)	175.84(3)	C(6)-C(7)-C(2)	118.9(3)
P(2)-Pd(1)-P(3)	84.37(3)	C(9)-C(8)-C(13)	120.9(2)
C(40)-Pd(2)-P(4)	84.95(9)	C(9)-C(8)-O(3)	121.6(2)
C(40)-Pd(2)-P(6)	92.16(9)	C(13)-C(8)-O(3)	117.5(2)
P(4)-Pd(2)-P(6)	176.08(3)	C(8)-C(9)-C(10)	118.6(3)
C(40)-Pd(2)-P(5)	175.79(9)	C(11)-C(10)-C(9)	121.8(3)
P(4)-Pd(2)-P(5)	98.36(3)	C(10)-C(11)-C(12)	118.9(3)
P(6)-Pd(2)-P(5)	84.65(3)	C(11)-C(12)-C(13)	120.4(3)
O(1)-P(1)-O(2)	109.88(10)	C(8)-C(13)-C(12)	119.2(3)
O(1)-P(1)-O(3)	104.83(10)	C(15)-C(14)-C(19)	118.7(2)
O(2)-P(1)-O(3)	100.33(10)	C(15)-C(14)-P(2)	122.3(2)
O(1)-P(1)-Pd(1)	119.65(8)	C(19)-C(14)-P(2)	118.9(2)
O(2)-P(1)-Pd(1)	107.96(7)	C(16)-C(15)-C(14)	120.4(3)
O(3)-P(1)-Pd(1)	112.48(7)	C(15)-C(16)-C(17)	120.2(3)
C(20)-P(2)-C(14)	107.16(13)	C(18)-C(17)-C(16)	119.9(3)
C(20)-P(2)-C(26)	104.58(12)	C(19)-C(18)-C(17)	120.0(3)
C(14)-P(2)-C(26)	102.03(12)	C(18)-C(19)-C(14)	120.9(3)
C(20)-P(2)-Pd(1)	111.37(9)	C(21)-C(20)-C(25)	119.0(2)
C(14)-P(2)-Pd(1)	122.02(9)	C(21)-C(20)-P(2)	118.8(2)
C(26)-P(2)-Pd(1)	108.02(9)	C(25)-C(20)-P(2)	122.1(2)
C(34)-P(3)-C(28)	108.98(12)	C(22)-C(21)-C(20)	120.3(3)
C(34)-P(3)-C(27)	103.00(12)	C(23)-C(22)-C(21)	120.0(3)
C(28)-P(3)-C(27)	102.05(12)	C(24)-C(23)-C(22)	120.4(3)
C(34)-P(3)-Pd(1)	110.27(9)	C(23)-C(24)-C(25)	120.3(3)
C(28)-P(3)-Pd(1)	122.94(9)	C(24)-C(25)-C(20)	120.1(3)
C(27)-P(3)-Pd(1)	107.48(9)	C(27)-C(26)-P(2)	109.50(18)
O(4)-P(4)-O(6)	109.22(11)	C(26)-C(27)-P(3)	109.96(18)
O(4)-P(4)-O(5)	104.47(11)	C(33)-C(28)-C(29)	118.8(3)
O(6)-P(4)-O(5)	100.98(10)	C(33)-C(28)-P(3)	120.0(2)
O(4)-P(4)-Pd(2)	119.17(8)	C(29)-C(28)-P(3)	121.1(2)
O(6)-P(4)-Pd(2)	108.75(7)	C(30)-C(29)-C(28)	120.4(3)
O(5)-P(4)-Pd(2)	112.69(7)	C(29)-C(30)-C(31)	119.8(3)
C(59)-P(5)-C(53)	105.86(12)	C(32)-C(31)-C(30)	119.8(3)
C(59)-P(5)-C(65)	105.77(12)	C(31)-C(32)-C(33)	120.9(3)
C(53)-P(5)-C(65)	102.39(12)	C(28)-C(33)-C(32)	120.3(3)
C(59)-P(5)-Pd(2)	114.35(9)	C(35)-C(34)-C(39)	118.7(2)
C(53)-P(5)-Pd(2)	119.44(9)	C(35)-C(34)-P(3)	123.0(2)
C(65)-P(5)-Pd(2)	107.64(9)	C(39)-C(34)-P(3)	118.04(19)
C(73)-P(6)-C(67)	105.17(12)	C(36)-C(35)-C(34)	120.3(3)
C(73)-P(6)-C(66)	104.73(12)	C(35)-C(36)-C(37)	120.3(3)
C(67)-P(6)-C(66)	104.48(12)	C(38)-C(37)-C(36)	120.2(3)
C(73)-P(6)-Pd(2)	109.11(9)	C(37)-C(38)-C(39)	119.4(3)
C(67)-P(6)-Pd(2)	123.72(9)	C(38)-C(39)-C(34)	121.2(2)
C(66)-P(6)-Pd(2)	108.10(9)	C(42)-C(41)-O(5)	122.9(2)
C(2)-O(2)-P(1)	123.28(17)	C(42)-C(41)-C(46)	120.3(3)
C(8)-O(3)-P(1)	122.37(16)	O(5)-C(41)-C(46)	116.7(2)
C(41)-O(5)-P(4)	123.62(16)	C(41)-C(42)-C(43)	119.5(3)
C(47)-O(6)-P(4)	123.38(16)	C(42)-C(43)-C(44)	120.4(3)
O(2)-C(2)-C(3)	120.6(2)	C(45)-C(44)-C(43)	119.3(3)
O(2)-C(2)-C(7)	118.8(2)	C(46)-C(45)-C(44)	120.8(3)
C(3)-C(2)-C(7)	120.4(3)	C(45)-C(46)-C(41)	119.6(3)

C(48)-C(47)-C(52)	120.3(3)	C(62)-C(63)-C(64)	120.4(3)
C(48)-C(47)-O(6)	119.0(3)	C(63)-C(64)-C(59)	119.9(3)
C(52)-C(47)-O(6)	120.6(2)	C(66)-C(65)-P(5)	110.12(18)
C(47)-C(48)-C(49)	117.9(3)	C(65)-C(66)-P(6)	109.08(18)
C(50)-C(49)-C(48)	121.2(3)	C(68)-C(67)-C(72)	118.8(2)
C(49)-C(50)-C(51)	121.1(3)	C(68)-C(67)-P(6)	121.1(2)
C(50)-C(51)-C(52)	119.3(4)	C(72)-C(67)-P(6)	119.9(2)
C(47)-C(52)-C(51)	120.3(3)	C(67)-C(68)-C(69)	120.5(3)
C(54)-C(53)-C(58)	118.8(2)	C(70)-C(69)-C(68)	120.5(3)
C(54)-C(53)-P(5)	118.6(2)	C(69)-C(70)-C(71)	119.5(3)
C(58)-C(53)-P(5)	122.4(2)	C(70)-C(71)-C(72)	120.9(3)
C(55)-C(54)-C(53)	120.9(3)	C(71)-C(72)-C(67)	119.7(3)
C(56)-C(55)-C(54)	120.0(3)	C(78)-C(73)-C(74)	119.1(2)
C(55)-C(56)-C(57)	119.5(3)	C(78)-C(73)-P(6)	119.1(2)
C(56)-C(57)-C(58)	121.0(3)	C(74)-C(73)-P(6)	121.8(2)
C(57)-C(58)-C(53)	119.8(3)	C(75)-C(74)-C(73)	120.3(3)
C(64)-C(59)-C(60)	118.9(2)	C(76)-C(75)-C(74)	120.6(3)
C(64)-C(59)-P(5)	122.0(2)	C(75)-C(76)-C(77)	120.0(3)
C(60)-C(59)-P(5)	119.1(2)	C(76)-C(77)-C(78)	119.7(3)
C(61)-C(60)-C(59)	120.3(2)	C(77)-C(78)-C(73)	120.4(3)
C(62)-C(61)-C(60)	120.8(3)	Cl(2)-C(79)-Cl(1)	110.73(19)
C(61)-C(62)-C(63)	119.6(3)	Cl(4)-C(80)-Cl(3)	110.8(2)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for compound **5**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Pd(1)	14(1)	14(1)	12(1)	2(1)	4(1)	2(1)
Pd(2)	15(1)	13(1)	11(1)	1(1)	4(1)	1(1)
Cl(1)	35(1)	34(1)	33(1)	10(1)	5(1)	-2(1)
Cl(2)	99(1)	38(1)	66(1)	-18(1)	36(1)	-16(1)
Cl(3)	27(1)	110(1)	46(1)	33(1)	1(1)	-17(1)
Cl(4)	36(1)	50(1)	63(1)	-16(1)	6(1)	-8(1)
P(1)	15(1)	16(1)	14(1)	3(1)	5(1)	3(1)
P(2)	15(1)	15(1)	13(1)	3(1)	5(1)	4(1)
P(3)	15(1)	15(1)	14(1)	1(1)	4(1)	2(1)
P(4)	17(1)	15(1)	14(1)	2(1)	5(1)	1(1)
P(5)	16(1)	13(1)	13(1)	2(1)	5(1)	2(1)
P(6)	16(1)	13(1)	13(1)	1(1)	4(1)	1(1)
O(1)	16(1)	21(1)	20(1)	3(1)	8(1)	4(1)
O(2)	20(1)	17(1)	20(1)	3(1)	9(1)	6(1)
O(3)	16(1)	23(1)	13(1)	-1(1)	5(1)	3(1)
O(4)	21(1)	19(1)	24(1)	8(1)	7(1)	3(1)
O(5)	22(1)	21(1)	12(1)	4(1)	4(1)	2(1)
O(6)	21(1)	18(1)	21(1)	6(1)	10(1)	3(1)
C(1)	22(1)	17(1)	19(1)	5(1)	6(1)	-2(1)
C(2)	24(1)	19(1)	14(1)	3(1)	7(1)	6(1)
C(3)	23(1)	21(1)	19(1)	2(1)	6(1)	6(1)
C(4)	29(2)	30(2)	21(2)	7(1)	8(1)	12(1)
C(5)	49(2)	20(2)	22(2)	3(1)	11(1)	14(1)
C(6)	39(2)	23(2)	28(2)	-4(1)	5(1)	0(1)
C(7)	25(1)	24(2)	25(2)	2(1)	6(1)	2(1)
C(8)	16(1)	17(1)	16(1)	-5(1)	3(1)	1(1)
C(9)	19(1)	24(2)	15(1)	-2(1)	3(1)	-2(1)
C(10)	20(1)	33(2)	20(1)	-7(1)	8(1)	-5(1)
C(11)	15(1)	27(2)	32(2)	-11(1)	7(1)	-1(1)
C(12)	19(1)	24(2)	27(2)	0(1)	-4(1)	4(1)
C(13)	24(1)	21(1)	20(1)	1(1)	7(1)	3(1)
C(14)	21(1)	17(1)	12(1)	4(1)	5(1)	2(1)
C(15)	22(1)	25(2)	17(1)	7(1)	7(1)	10(1)
C(16)	31(2)	19(1)	25(2)	4(1)	8(1)	6(1)
C(17)	29(2)	21(2)	21(2)	5(1)	4(1)	-1(1)
C(18)	20(1)	34(2)	21(1)	5(1)	4(1)	2(1)
C(19)	22(1)	20(1)	21(1)	4(1)	6(1)	4(1)
C(20)	16(1)	18(1)	17(1)	1(1)	4(1)	5(1)
C(21)	20(1)	34(2)	18(1)	6(1)	6(1)	6(1)
C(22)	21(1)	43(2)	30(2)	5(1)	10(1)	8(1)
C(23)	20(1)	35(2)	25(2)	2(1)	1(1)	11(1)
C(24)	25(2)	32(2)	19(1)	5(1)	2(1)	10(1)
C(25)	22(1)	21(1)	19(1)	2(1)	6(1)	5(1)
C(26)	20(1)	21(1)	16(1)	6(1)	8(1)	6(1)
C(27)	20(1)	20(1)	14(1)	2(1)	5(1)	4(1)
C(28)	13(1)	15(1)	20(1)	1(1)	1(1)	2(1)
C(29)	28(2)	24(2)	21(1)	0(1)	5(1)	1(1)
C(30)	29(2)	20(2)	27(2)	-5(1)	4(1)	1(1)
C(31)	20(1)	20(2)	36(2)	2(1)	2(1)	-2(1)
C(32)	26(2)	20(2)	32(2)	6(1)	12(1)	1(1)
C(33)	22(1)	22(1)	20(1)	1(1)	5(1)	4(1)

C(34)	15(1)	14(1)	16(1)	6(1)	4(1)	4(1)
C(35)	20(1)	17(1)	22(1)	0(1)	4(1)	3(1)
C(36)	23(1)	20(1)	28(2)	3(1)	11(1)	10(1)
C(37)	16(1)	27(2)	30(2)	6(1)	8(1)	4(1)
C(38)	19(1)	28(2)	23(2)	0(1)	1(1)	0(1)
C(39)	21(1)	20(1)	18(1)	-1(1)	7(1)	5(1)
C(40)	24(2)	23(2)	14(1)	0(1)	8(1)	6(1)
C(41)	16(1)	21(1)	13(1)	5(1)	-3(1)	3(1)
C(42)	22(1)	23(2)	15(1)	0(1)	0(1)	1(1)
C(43)	23(1)	31(2)	24(2)	7(1)	3(1)	2(1)
C(44)	21(1)	22(2)	31(2)	3(1)	-3(1)	-5(1)
C(45)	31(2)	27(2)	28(2)	-1(1)	-1(1)	6(1)
C(46)	28(2)	28(2)	18(1)	0(1)	3(1)	7(1)
C(47)	24(1)	23(2)	16(1)	4(1)	9(1)	13(1)
C(48)	24(2)	59(2)	28(2)	15(2)	9(1)	13(2)
C(49)	40(2)	83(3)	37(2)	35(2)	23(2)	44(2)
C(50)	74(3)	39(2)	39(2)	22(2)	39(2)	38(2)
C(51)	69(2)	24(2)	26(2)	5(1)	17(2)	13(2)
C(52)	40(2)	23(2)	21(2)	5(1)	5(1)	7(1)
C(53)	17(1)	20(1)	14(1)	-1(1)	6(1)	2(1)
C(54)	22(1)	19(1)	19(1)	4(1)	7(1)	0(1)
C(55)	20(1)	30(2)	23(2)	-3(1)	7(1)	-6(1)
C(56)	18(1)	35(2)	33(2)	2(1)	7(1)	6(1)
C(57)	22(1)	23(2)	42(2)	2(1)	3(1)	7(1)
C(58)	18(1)	17(1)	26(2)	0(1)	5(1)	-1(1)
C(59)	15(1)	14(1)	15(1)	1(1)	2(1)	2(1)
C(60)	16(1)	19(1)	17(1)	3(1)	5(1)	2(1)
C(61)	18(1)	21(1)	24(2)	5(1)	5(1)	1(1)
C(62)	20(1)	18(1)	26(2)	2(1)	0(1)	3(1)
C(63)	27(2)	23(2)	20(1)	-3(1)	3(1)	4(1)
C(64)	21(1)	20(1)	19(1)	3(1)	5(1)	4(1)
C(65)	24(1)	15(1)	15(1)	4(1)	11(1)	4(1)
C(66)	22(1)	15(1)	15(1)	2(1)	3(1)	3(1)
C(67)	18(1)	18(1)	17(1)	6(1)	7(1)	6(1)
C(68)	24(1)	20(1)	22(2)	-1(1)	4(1)	7(1)
C(69)	22(1)	27(2)	24(2)	3(1)	-2(1)	4(1)
C(70)	19(1)	23(2)	33(2)	8(1)	8(1)	7(1)
C(71)	22(1)	17(2)	41(2)	-4(1)	13(1)	5(1)
C(72)	19(1)	21(2)	27(2)	-5(1)	5(1)	0(1)
C(73)	17(1)	10(1)	23(1)	-2(1)	8(1)	2(1)
C(74)	20(1)	21(1)	28(2)	5(1)	8(1)	4(1)
C(75)	25(2)	17(1)	38(2)	8(1)	12(1)	6(1)
C(76)	17(1)	19(2)	42(2)	-2(1)	11(1)	-1(1)
C(77)	18(1)	24(2)	27(2)	-4(1)	6(1)	1(1)
C(78)	19(1)	18(1)	17(1)	2(1)	7(1)	2(1)
C(79)	30(2)	35(2)	30(2)	4(1)	6(1)	2(1)
C(80)	35(2)	32(2)	38(2)	8(2)	12(1)	-3(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for compound **5**.

	x	y	z	U(eq)
H(1A)	4890	2972	4103	29
H(1B)	3373	3380	4194	29
H(1C)	3330	2306	3582	29
H(3)	6928	-1302	4433	24
H(4)	7735	-2960	3959	31
H(5)	6012	-4589	3393	35
H(6)	3507	-4559	3281	38
H(7)	2668	-2907	3725	30
H(9)	1411	-745	4197	24
H(10)	-1038	-472	4129	30
H(11)	-2384	489	3130	30
H(12)	-1298	1132	2142	30
H(13)	1175	900	2210	26
H(15)	4220	-2115	5801	24
H(16)	2616	-3860	5485	29
H(17)	68	-3878	5344	29
H(18)	-879	-2138	5472	30
H(19)	701	-391	5735	25
H(21)	6611	743	5627	28
H(22)	9058	502	6253	36
H(23)	9582	-336	7445	33
H(24)	7703	-863	8047	31
H(25)	5252	-635	7433	25
H(26A)	2076	984	6608	21
H(26B)	3511	720	7257	21
H(27A)	4884	2443	7100	21
H(27B)	3336	2772	7207	21
H(29)	4260	4692	7236	30
H(30)	5794	6477	7555	32
H(31)	7061	7216	6660	32
H(32)	6700	6220	5443	30
H(33)	5147	4449	5106	26
H(35)	1590	4455	6501	24
H(36)	-967	4477	6260	27
H(37)	-2632	3149	5305	28
H(38)	-1743	1799	4579	29
H(39)	818	1787	4810	23
H(40A)	4893	7388	-1108	29
H(40B)	6426	7372	-456	29
H(40C)	6252	8393	-1031	29
H(42)	1445	9126	-855	25
H(43)	-436	7566	-967	32
H(44)	-830	6052	-1958	32
H(45)	654	6115	-2835	36
H(46)	2576	7633	-2704	30
H(48)	1083	11536	-1650	43
H(49)	743	13384	-2110	56
H(50)	2627	14883	-1778	52
H(51)	4912	14660	-960	46
H(52)	5283	12866	-464	34

H(54)	1281	8355	712	24
H(55)	-1221	8573	278	30
H(56)	-1906	10400	17	34
H(57)	-86	12012	239	35
H(58)	2425	11813	682	25
H(60)	6161	11424	539	20
H(61)	7496	13235	1034	25
H(62)	7348	14104	2223	27
H(63)	5852	13144	2935	29
H(64)	4429	11350	2427	24
H(65A)	4162	9388	2310	20
H(65B)	3254	8308	1711	20
H(66A)	5504	7714	2356	21
H(66B)	6436	8882	2199	21
H(68)	8505	8285	2177	27
H(69)	10820	7643	2420	31
H(70)	11188	6095	1643	29
H(71)	9198	5111	662	31
H(72)	6863	5726	411	28
H(74)	5225	5471	1788	27
H(75)	3411	3853	1517	31
H(76)	1359	3731	434	31
H(77)	1116	5237	-398	28
H(78)	2951	6859	-141	21
H(79A)	-760	3045	3139	39
H(79B)	920	3065	3648	39
H(80A)	6867	2122	8413	42
H(80B)	8614	2095	8555	42

Table 6. Torsion angles [°] for compound **5**.

C(1)-Pd(1)-P(1)-O(1)	61.22(12)
P(2)-Pd(1)-P(1)-O(1)	-117.52(10)
P(3)-Pd(1)-P(1)-O(1)	-2.7(4)
C(1)-Pd(1)-P(1)-O(2)	-172.21(11)
P(2)-Pd(1)-P(1)-O(2)	9.05(8)
P(3)-Pd(1)-P(1)-O(2)	123.9(4)
C(1)-Pd(1)-P(1)-O(3)	-62.44(11)
P(2)-Pd(1)-P(1)-O(3)	118.82(8)
P(3)-Pd(1)-P(1)-O(3)	-126.4(4)
C(1)-Pd(1)-P(2)-C(20)	-30(4)
P(1)-Pd(1)-P(2)-C(20)	72.56(10)
P(3)-Pd(1)-P(2)-C(20)	-103.65(9)
C(1)-Pd(1)-P(2)-C(14)	-159(4)
P(1)-Pd(1)-P(2)-C(14)	-55.63(11)
P(3)-Pd(1)-P(2)-C(14)	128.16(11)
C(1)-Pd(1)-P(2)-C(26)	84(4)
P(1)-Pd(1)-P(2)-C(26)	-173.14(10)
P(3)-Pd(1)-P(2)-C(26)	10.66(10)
C(1)-Pd(1)-P(3)-C(34)	82.44(12)
P(1)-Pd(1)-P(3)-C(34)	146.0(4)
P(2)-Pd(1)-P(3)-C(34)	-98.80(9)
C(1)-Pd(1)-P(3)-C(28)	-48.29(13)
P(1)-Pd(1)-P(3)-C(28)	15.2(4)
P(2)-Pd(1)-P(3)-C(28)	130.48(11)
C(1)-Pd(1)-P(3)-C(27)	-165.99(12)
P(1)-Pd(1)-P(3)-C(27)	-102.5(4)
P(2)-Pd(1)-P(3)-C(27)	12.78(9)
C(40)-Pd(2)-P(4)-O(4)	-66.47(13)
P(6)-Pd(2)-P(4)-O(4)	-23.8(4)
P(5)-Pd(2)-P(4)-O(4)	116.16(10)
C(40)-Pd(2)-P(4)-O(6)	167.55(11)
P(6)-Pd(2)-P(4)-O(6)	-149.8(4)
P(5)-Pd(2)-P(4)-O(6)	-9.82(8)
C(40)-Pd(2)-P(4)-O(5)	56.46(11)
P(6)-Pd(2)-P(4)-O(5)	99.1(4)
P(5)-Pd(2)-P(4)-O(5)	-120.91(8)
C(40)-Pd(2)-P(5)-C(59)	149.2(12)
P(4)-Pd(2)-P(5)-C(59)	-69.24(10)
P(6)-Pd(2)-P(5)-C(59)	108.23(10)
C(40)-Pd(2)-P(5)-C(53)	-83.9(12)
P(4)-Pd(2)-P(5)-C(53)	57.61(11)
P(6)-Pd(2)-P(5)-C(53)	-124.92(11)
C(40)-Pd(2)-P(5)-C(65)	32.1(12)
P(4)-Pd(2)-P(5)-C(65)	173.58(10)
P(6)-Pd(2)-P(5)-C(65)	-8.95(10)
C(40)-Pd(2)-P(6)-C(73)	-78.53(12)
P(4)-Pd(2)-P(6)-C(73)	-121.0(4)
P(5)-Pd(2)-P(6)-C(73)	98.71(9)
C(40)-Pd(2)-P(6)-C(67)	45.82(14)
P(4)-Pd(2)-P(6)-C(67)	3.3(4)
P(5)-Pd(2)-P(6)-C(67)	-136.94(11)
C(40)-Pd(2)-P(6)-C(66)	168.12(12)
P(4)-Pd(2)-P(6)-C(66)	125.7(4)
P(5)-Pd(2)-P(6)-C(66)	-14.64(10)
O(1)-P(1)-O(2)-C(2)	-37.0(2)
O(3)-P(1)-O(2)-C(2)	73.1(2)

Pd(1)-P(1)-O(2)-C(2)	-169.04(18)
O(1)-P(1)-O(3)-C(8)	-160.0(2)
O(2)-P(1)-O(3)-C(8)	86.0(2)
Pd(1)-P(1)-O(3)-C(8)	-28.5(2)
O(4)-P(4)-O(5)-C(41)	165.66(19)
O(6)-P(4)-O(5)-C(41)	-81.0(2)
Pd(2)-P(4)-O(5)-C(41)	34.9(2)
O(4)-P(4)-O(6)-C(47)	29.4(2)
O(5)-P(4)-O(6)-C(47)	-80.3(2)
Pd(2)-P(4)-O(6)-C(47)	161.01(18)
P(1)-O(2)-C(2)-C(3)	68.0(3)
P(1)-O(2)-C(2)-C(7)	-116.8(2)
O(2)-C(2)-C(3)-C(4)	175.7(2)
C(7)-C(2)-C(3)-C(4)	0.6(4)
C(2)-C(3)-C(4)-C(5)	-0.9(4)
C(3)-C(4)-C(5)-C(6)	0.5(5)
C(4)-C(5)-C(6)-C(7)	0.2(5)
C(5)-C(6)-C(7)-C(2)	-0.5(5)
O(2)-C(2)-C(7)-C(6)	-175.1(3)
C(3)-C(2)-C(7)-C(6)	0.1(4)
P(1)-O(3)-C(8)-C(9)	-43.1(3)
P(1)-O(3)-C(8)-C(13)	138.1(2)
C(13)-C(8)-C(9)-C(10)	-4.3(4)
O(3)-C(8)-C(9)-C(10)	177.0(2)
C(8)-C(9)-C(10)-C(11)	1.6(4)
C(9)-C(10)-C(11)-C(12)	1.6(4)
C(10)-C(11)-C(12)-C(13)	-2.2(4)
C(9)-C(8)-C(13)-C(12)	3.7(4)
O(3)-C(8)-C(13)-C(12)	-177.5(2)
C(11)-C(12)-C(13)-C(8)	-0.4(4)
C(20)-P(2)-C(14)-C(15)	-25.9(3)
C(26)-P(2)-C(14)-C(15)	-135.5(2)
Pd(1)-P(2)-C(14)-C(15)	104.1(2)
C(20)-P(2)-C(14)-C(19)	157.9(2)
C(26)-P(2)-C(14)-C(19)	48.3(2)
Pd(1)-P(2)-C(14)-C(19)	-72.1(2)
C(19)-C(14)-C(15)-C(16)	0.2(4)
P(2)-C(14)-C(15)-C(16)	-176.0(2)
C(14)-C(15)-C(16)-C(17)	-1.5(4)
C(15)-C(16)-C(17)-C(18)	1.3(4)
C(16)-C(17)-C(18)-C(19)	0.1(4)
C(17)-C(18)-C(19)-C(14)	-1.3(4)
C(15)-C(14)-C(19)-C(18)	1.2(4)
P(2)-C(14)-C(19)-C(18)	177.5(2)
C(14)-P(2)-C(20)-C(21)	123.2(2)
C(26)-P(2)-C(20)-C(21)	-129.0(2)
Pd(1)-P(2)-C(20)-C(21)	-12.6(2)
C(14)-P(2)-C(20)-C(25)	-59.9(3)
C(26)-P(2)-C(20)-C(25)	47.9(3)
Pd(1)-P(2)-C(20)-C(25)	164.4(2)
C(25)-C(20)-C(21)-C(22)	0.6(4)
P(2)-C(20)-C(21)-C(22)	177.7(2)
C(20)-C(21)-C(22)-C(23)	0.3(5)
C(21)-C(22)-C(23)-C(24)	-1.4(5)
C(22)-C(23)-C(24)-C(25)	1.5(5)
C(23)-C(24)-C(25)-C(20)	-0.5(4)

C(21)-C(20)-C(25)-C(24)	-0.6(4)
P(2)-C(20)-C(25)-C(24)	-177.6(2)
C(20)-P(2)-C(26)-C(27)	80.6(2)
C(14)-P(2)-C(26)-C(27)	-167.88(18)
Pd(1)-P(2)-C(26)-C(27)	-38.1(2)
P(2)-C(26)-C(27)-P(3)	50.3(2)
C(34)-P(3)-C(27)-C(26)	76.3(2)
C(28)-P(3)-C(27)-C(26)	-170.69(18)
Pd(1)-P(3)-C(27)-C(26)	-40.13(19)
C(34)-P(3)-C(28)-C(33)	-108.9(2)
C(27)-P(3)-C(28)-C(33)	142.7(2)
Pd(1)-P(3)-C(28)-C(33)	22.4(3)
C(34)-P(3)-C(28)-C(29)	75.7(2)
C(27)-P(3)-C(28)-C(29)	-32.7(2)
Pd(1)-P(3)-C(28)-C(29)	-153.03(19)
C(33)-C(28)-C(29)-C(30)	0.4(4)
P(3)-C(28)-C(29)-C(30)	175.9(2)
C(28)-C(29)-C(30)-C(31)	-1.2(4)
C(29)-C(30)-C(31)-C(32)	1.5(5)
C(30)-C(31)-C(32)-C(33)	-1.0(5)
C(29)-C(28)-C(33)-C(32)	0.0(4)
P(3)-C(28)-C(33)-C(32)	-175.5(2)
C(31)-C(32)-C(33)-C(28)	0.2(4)
C(28)-P(3)-C(34)-C(35)	-39.9(3)
C(27)-P(3)-C(34)-C(35)	67.9(2)
Pd(1)-P(3)-C(34)-C(35)	-177.6(2)
C(28)-P(3)-C(34)-C(39)	145.8(2)
C(27)-P(3)-C(34)-C(39)	-106.3(2)
Pd(1)-P(3)-C(34)-C(39)	8.1(2)
C(39)-C(34)-C(35)-C(36)	0.0(4)
P(3)-C(34)-C(35)-C(36)	-174.2(2)
C(34)-C(35)-C(36)-C(37)	0.3(4)
C(35)-C(36)-C(37)-C(38)	-0.3(5)
C(36)-C(37)-C(38)-C(39)	-0.1(4)
C(37)-C(38)-C(39)-C(34)	0.4(4)
C(35)-C(34)-C(39)-C(38)	-0.3(4)
P(3)-C(34)-C(39)-C(38)	174.2(2)
P(4)-O(5)-C(41)-C(42)	40.1(3)
P(4)-O(5)-C(41)-C(46)	-142.5(2)
O(5)-C(41)-C(42)-C(43)	179.1(2)
C(46)-C(41)-C(42)-C(43)	1.7(4)
C(41)-C(42)-C(43)-C(44)	-1.3(4)
C(42)-C(43)-C(44)-C(45)	-0.2(4)
C(43)-C(44)-C(45)-C(46)	1.3(5)
C(44)-C(45)-C(46)-C(41)	-0.9(5)
C(42)-C(41)-C(46)-C(45)	-0.7(4)
O(5)-C(41)-C(46)-C(45)	-178.2(3)
P(4)-O(6)-C(47)-C(48)	110.9(3)
P(4)-O(6)-C(47)-C(52)	-73.2(3)
C(52)-C(47)-C(48)-C(49)	-1.3(4)
O(6)-C(47)-C(48)-C(49)	174.6(3)
C(47)-C(48)-C(49)-C(50)	1.5(5)
C(48)-C(49)-C(50)-C(51)	-0.6(5)
C(49)-C(50)-C(51)-C(52)	-0.3(5)
C(48)-C(47)-C(52)-C(51)	0.5(5)
O(6)-C(47)-C(52)-C(51)	-175.4(3)

C(50)-C(51)-C(52)-C(47)	0.4(5)
C(59)-P(5)-C(53)-C(54)	-161.8(2)
C(65)-P(5)-C(53)-C(54)	-51.3(2)
Pd(2)-P(5)-C(53)-C(54)	67.4(2)
C(59)-P(5)-C(53)-C(58)	23.7(3)
C(65)-P(5)-C(53)-C(58)	134.3(2)
Pd(2)-P(5)-C(53)-C(58)	-107.0(2)
C(58)-C(53)-C(54)-C(55)	0.0(4)
P(5)-C(53)-C(54)-C(55)	-174.6(2)
C(53)-C(54)-C(55)-C(56)	0.7(4)
C(54)-C(55)-C(56)-C(57)	-1.2(5)
C(55)-C(56)-C(57)-C(58)	0.9(5)
C(56)-C(57)-C(58)-C(53)	-0.2(5)
C(54)-C(53)-C(58)-C(57)	-0.3(4)
P(5)-C(53)-C(58)-C(57)	174.1(2)
C(53)-P(5)-C(59)-C(64)	67.0(2)
C(65)-P(5)-C(59)-C(64)	-41.2(2)
Pd(2)-P(5)-C(59)-C(64)	-159.42(19)
C(53)-P(5)-C(59)-C(60)	-113.2(2)
C(65)-P(5)-C(59)-C(60)	138.6(2)
Pd(2)-P(5)-C(59)-C(60)	20.4(2)
C(64)-C(59)-C(60)-C(61)	-1.8(4)
P(5)-C(59)-C(60)-C(61)	178.4(2)
C(59)-C(60)-C(61)-C(62)	1.4(4)
C(60)-C(61)-C(62)-C(63)	0.3(4)
C(61)-C(62)-C(63)-C(64)	-1.5(4)
C(62)-C(63)-C(64)-C(59)	1.1(4)
C(60)-C(59)-C(64)-C(63)	0.6(4)
P(5)-C(59)-C(64)-C(63)	-179.6(2)
C(59)-P(5)-C(65)-C(66)	-86.13(19)
C(53)-P(5)-C(65)-C(66)	163.22(18)
Pd(2)-P(5)-C(65)-C(66)	36.50(19)
P(5)-C(65)-C(66)-P(6)	-49.7(2)
C(73)-P(6)-C(66)-C(65)	-75.2(2)
C(67)-P(6)-C(66)-C(65)	174.43(18)
Pd(2)-P(6)-C(66)-C(65)	41.0(2)
C(73)-P(6)-C(67)-C(68)	-140.8(2)
C(66)-P(6)-C(67)-C(68)	-30.8(3)
Pd(2)-P(6)-C(67)-C(68)	93.1(2)
C(73)-P(6)-C(67)-C(72)	44.2(3)
C(66)-P(6)-C(67)-C(72)	154.2(2)
Pd(2)-P(6)-C(67)-C(72)	-81.9(2)
C(72)-C(67)-C(68)-C(69)	-0.7(4)
P(6)-C(67)-C(68)-C(69)	-175.8(2)
C(67)-C(68)-C(69)-C(70)	1.3(5)
C(68)-C(69)-C(70)-C(71)	-1.7(5)
C(69)-C(70)-C(71)-C(72)	1.5(5)
C(70)-C(71)-C(72)-C(67)	-0.9(5)
C(68)-C(67)-C(72)-C(71)	0.5(4)
P(6)-C(67)-C(72)-C(71)	175.6(2)
C(67)-P(6)-C(73)-C(78)	-135.6(2)
C(66)-P(6)-C(73)-C(78)	114.5(2)
Pd(2)-P(6)-C(73)-C(78)	-1.0(2)
C(67)-P(6)-C(73)-C(74)	45.9(2)
C(66)-P(6)-C(73)-C(74)	-64.0(2)
Pd(2)-P(6)-C(73)-C(74)	-179.50(19)

C(78)-C(73)-C(74)-C(75)	0.7(4)
P(6)-C(73)-C(74)-C(75)	179.2(2)
C(73)-C(74)-C(75)-C(76)	-0.7(4)
C(74)-C(75)-C(76)-C(77)	0.2(4)
C(75)-C(76)-C(77)-C(78)	0.3(4)
C(76)-C(77)-C(78)-C(73)	-0.3(4)
C(74)-C(73)-C(78)-C(77)	-0.2(4)
P(6)-C(73)-C(78)-C(77)	-178.8(2)

Symmetry transformations used to generate equivalent atoms:

Crystallographic Experimental Section and discussion for compound 9.

Data Collection

An air-sensitive crystal with approximate dimensions 0.39 x 0.25 x 0.18 mm^3 was selected under oil under ambient conditions and attached to the tip of a glass capillary. The crystal was mounted in a stream of cold nitrogen at 100(2) K and centered in the X-ray beam by using a video camera.

The crystal evaluation and data collection were performed on a Bruker CCD-1000 diffractometer with Mo K_α (λ = 0.71073 Å) radiation and the diffractometer to crystal distance of 4.9 cm.

The initial cell constants were obtained from three series of ω scans at different starting angles. Each series consisted of 30 frames collected at intervals of 0.3º in a 6º range about ω with the exposure time of 15 seconds per frame. A total of 218 reflections was obtained. The reflections were successfully indexed by an automated indexing routine built in the SMART program. The final cell constants were calculated from a set of 13025 strong reflections from the actual data collection.

The data were collected by using the hemisphere data collection routine. The reciprocal space was surveyed to the extent of a full sphere to a resolution of 0.80 Å. A total of 29205 data were harvested by collecting three sets of frames with 0.3º scans in ω with an exposure time 70 s per frame. These highly redundant datasets were corrected for Lorentz and polarization effects. The absorption correction was based on fitting a function to the empirical transmission surface as sampled by multiple equivalent measurements.[1]

Structure Solution and Refinement

The systematic absences in the diffraction data were uniquely consistent for the space group $P2_1/c$ that yielded chemically reasonable and computationally stable results of refinement.[1]

A successful solution by the direct methods provided most non-hydrogen atoms from the *E*-map. The remaining non-hydrogen atoms were located in an alternating series of least-squares cycles and difference Fourier maps. All non-hydrogen atoms were refined with anisotropic displacement coefficients. All hydrogen atoms were included in the structure factor calculation at idealized positions and were allowed to ride on the neighboring atoms with relative isotropic

displacement coefficients. The Pd complex occupies a crystallographic inversion center. There is also one solvate molecule of dichloromethane in the asymmetric unit.

The final least-squares refinement of 439 parameters against 7400 data resulted in residuals *R* (based on F^2 for $I \geq 2\sigma$) and *wR* (based on F^2 for all data) of 0.0662 and 0.1511, respectively. The final difference Fourier map was featureless.

The ORTEP diagrams are drawn with 50% probability ellipsoids.

A Cambridge Structural Database (CSD) search returned three molecules relevant to compound **9**.[3] Two of these molecules have a square planar geometry, $[Pd(dppe)_2][SO_3CF_3]_2$ (**10**)[4] and $[Pd(dppe)_2]Cl_2$ (**11**)[5] while the third has a tetrahedral geometry, [Pd(dpce)(dppe)] (dcpe = 1,2-bis(dicyclohexylphosphino)ethane) (**12**).[6] The average Pd—P distance in **9** is slightly shorter than that in **10** and **11** (2.356(9)Å); however the difference is not statistically significant. The average ligand bite angle in **9** is very similar to that in **10** and **11** (82.4(6)˚). Thus, the geometry about the Pd(II) center in **9** is typical.

The tetrahedral Pd(0) ce[7]nter in **12** is similar to **9**. The Pd—P distances in **9** and **12** ((av.)2.343(17)Å) are essentially equal. However, the average ligand bite angle in **9** is appreciably smaller than that in **12** (86.3(9)˚), while the average of the other P–Pd–P angles in **9** (97.34(4)˚) is substantially smaller than corresponding angles in **12** (122(2)˚). Therefore, while the Pd—P distances in **9** compare well to the literature values, the angles in the square planar complex expectedly differ from those in the tetrahedral molecule.

The anion $[PO_2(OPh)_2]^-$ has a distorted tetrahedral geometry. The P—O(oxo) and P—OPh distances in **9** are (av.)1.469(8)Å and (av.)1.630(3)Å, respectively, while the O–P–O and PhO–P–OPh angles average 121.5(2)˚ and 102.44(18)˚, correspondingly. A CSD search revealed nine identical anions. The P—O(oxo) ((av.)1.479(12)Å) and P—OPh ((av.)1.601(14)Å) distances and O–P–O (av.)(120.1(16)˚) and PhO–P–OPh (av.)(102(3)˚) angles were statistically similar to the corresponding values in **9**; therefore $[PO_2(OPh)_2]^-$ is typical as compared to the literature data.

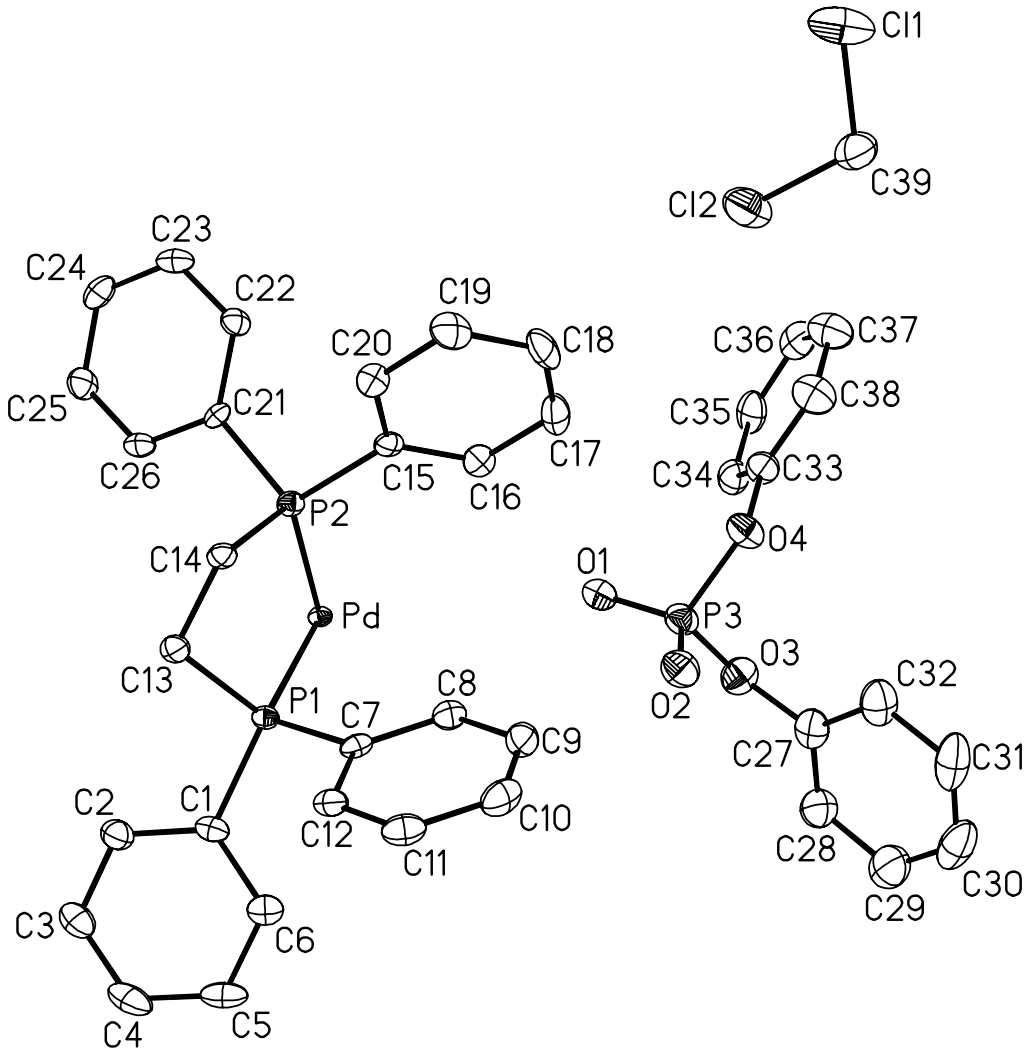

Figure 1. The content of the asymmetric unit. The H atoms are omitted for clarity.

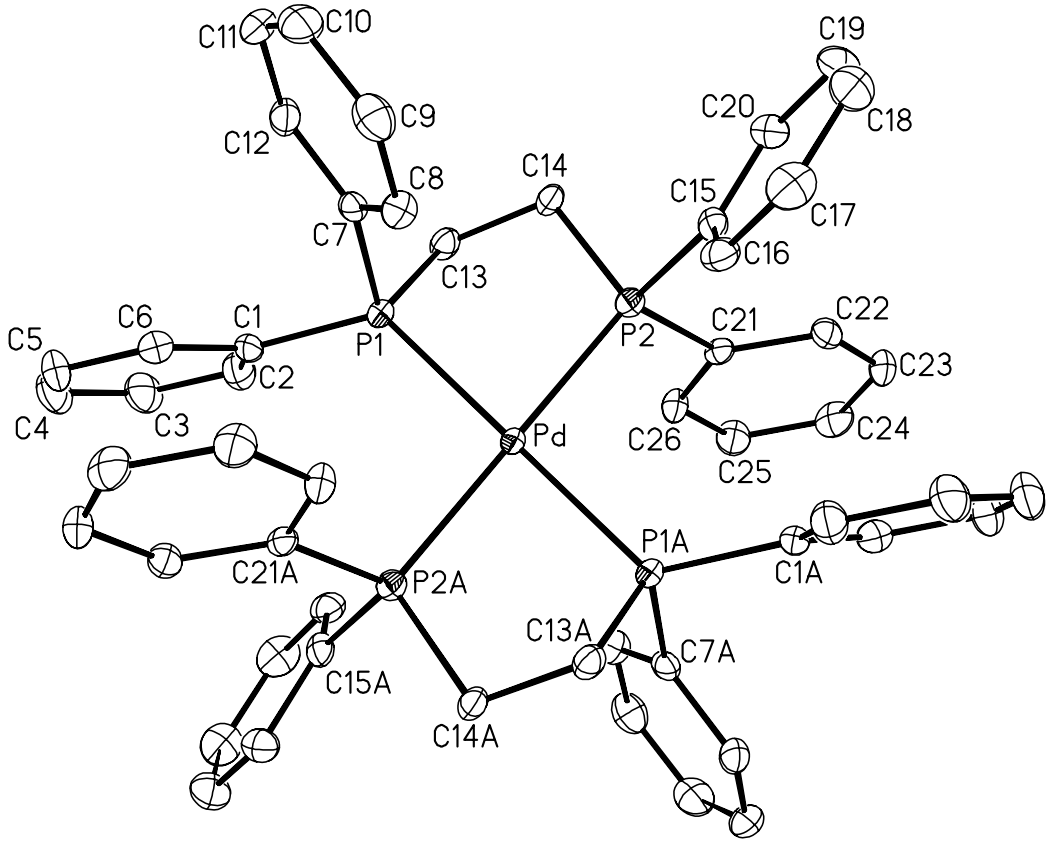

Figure 2. The Pd complex. The H atoms are omitted for clarity.

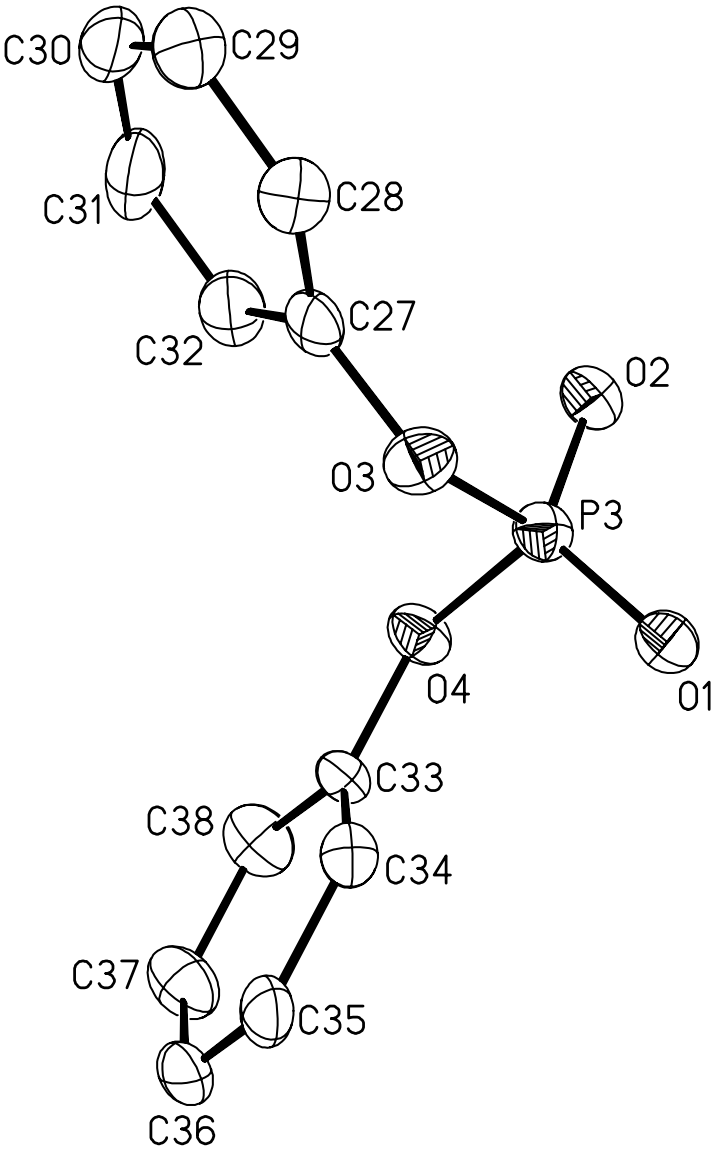

Figure 3. The counterion. The H atoms are omitted for clarity.

Table 1. Crystal data and structure refinement for compound **9**.

Identification code	sto05	
Empirical formula	$C_{78}\ H_{72}\ Cl_4\ O_8\ P_6\ Pd$	
Formula weight	1571.38	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 12.5532(15) Å	α= 90°.
	b = 16.599(2) Å	β= 99.375(2)°.
	c = 17.600(2) Å	γ = 90°.
Volume	3618.2(8) $Å^3$	
Z	2	
Density (calculated)	1.442 Mg/m^3	
Absorption coefficient	0.593 mm^{-1}	
F(000)	1616	
Crystal size	0.39 x 0.25 x 0.18 mm^3	
Theta range for data collection	2.35 to 26.46°.	
Index ranges	-15<=h<=15, -20<=k<=20, -22<=l<=22	
Reflections collected	29205	
Independent reflections	7400 [R(int) = 0.0572]	
Completeness to theta = 26.46°	99.1 %	
Absorption correction	Multiscan with SADABS	
Max. and min. transmission	0.9007 and 0.8016	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7400 / 0 / 439	
Goodness-of-fit on F^2	1.118	
Final R indices [I>2sigma(I)]	R1 = 0.0662, wR2 = 0.1441	
R indices (all data)	R1 = 0.0853, wR2 = 0.1511	
Largest diff. peak and hole	1.020 and -1.076 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for compound **9**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Pd	5000	0	0	11(1)
Cl(1)	7767(2)	682(1)	-5970(1)	53(1)
Cl(2)	7662(1)	651(1)	-4329(1)	44(1)
P(1)	3869(1)	1068(1)	230(1)	13(1)
P(2)	3964(1)	48(1)	-1228(1)	15(1)
P(3)	8503(1)	1470(1)	-768(1)	25(1)
O(1)	8008(3)	723(2)	-553(2)	32(1)
O(2)	7912(3)	2241(2)	-823(2)	31(1)
O(3)	9633(3)	1568(2)	-169(2)	32(1)
O(4)	8916(3)	1374(2)	-1593(2)	28(1)
C(1)	3546(3)	1330(3)	1167(2)	17(1)
C(2)	2684(4)	951(3)	1438(3)	23(1)
C(3)	2425(4)	1171(3)	2150(3)	28(1)
C(4)	3005(4)	1766(3)	2578(3)	33(1)
C(5)	3857(4)	2144(3)	2316(3)	30(1)
C(6)	4127(4)	1928(3)	1610(3)	21(1)
C(7)	4302(3)	2019(2)	-137(2)	16(1)
C(8)	5261(4)	2085(3)	-424(2)	20(1)
C(9)	5568(4)	2826(3)	-691(3)	25(1)
C(10)	4911(4)	3495(3)	-671(3)	28(1)
C(11)	3948(4)	3430(3)	-394(3)	24(1)
C(12)	3637(4)	2693(3)	-125(2)	19(1)
C(13)	2583(3)	839(3)	-383(2)	17(1)
C(14)	2800(3)	726(2)	-1203(2)	16(1)
C(15)	4572(3)	428(2)	-2021(2)	16(1)
C(16)	5668(4)	598(3)	-1930(3)	20(1)
C(17)	6106(4)	896(3)	-2547(3)	28(1)
C(18)	5454(4)	1025(3)	-3249(3)	29(1)
C(19)	4363(4)	861(3)	-3340(3)	28(1)
C(20)	3917(4)	570(3)	-2728(2)	21(1)
C(21)	3393(3)	-932(2)	-1517(2)	13(1)
C(22)	3447(4)	-1285(3)	-2224(2)	18(1)
C(23)	2951(4)	-2022(3)	-2415(3)	23(1)
C(24)	2421(4)	-2416(3)	-1897(3)	24(1)
C(25)	2383(4)	-2071(3)	-1184(3)	23(1)
C(26)	2862(4)	-1337(3)	-993(2)	19(1)
C(27)	10190(4)	2290(3)	-77(3)	30(1)
C(28)	10642(4)	2500(3)	669(3)	37(1)
C(29)	11203(5)	3224(4)	810(4)	47(2)
C(30)	11322(5)	3720(3)	196(4)	49(2)
C(31)	10891(4)	3498(3)	-554(4)	47(2)
C(32)	10324(4)	2786(3)	-698(4)	38(1)
C(33)	9499(4)	720(3)	-1803(3)	26(1)
C(34)	9863(4)	92(3)	-1306(3)	28(1)
C(35)	10403(4)	-541(3)	-1595(3)	32(1)
C(36)	10591(4)	-544(3)	-2345(3)	35(1)
C(37)	10259(4)	99(3)	-2814(3)	39(1)
C(38)	9715(4)	733(3)	-2550(3)	36(1)
C(39)	8286(5)	1074(3)	-5064(3)	38(1)

Table 3. Bond lengths [Å] and angles [°] for compound **9**.

Pd-P(2)#1	2.3348(10)	C(15)-C(20)	1.394(6)
Pd-P(2)	2.3348(10)	C(16)-C(17)	1.386(6)
Pd-P(1)	2.3469(10)	C(16)-H(16A)	0.9500
Pd-P(1)#1	2.3469(10)	C(17)-C(18)	1.382(7)
Cl(1)-C(39)	1.747(5)	C(17)-H(17A)	0.9500
Cl(2)-C(39)	1.764(6)	C(18)-C(19)	1.380(7)
P(1)-C(1)	1.814(4)	C(18)-H(18A)	0.9500
P(1)-C(7)	1.821(4)	C(19)-C(20)	1.380(6)
P(1)-C(13)	1.830(4)	C(19)-H(19A)	0.9500
P(2)-C(15)	1.810(4)	C(20)-H(20A)	0.9500
P(2)-C(21)	1.818(4)	C(21)-C(22)	1.387(6)
P(2)-C(14)	1.851(4)	C(21)-C(26)	1.396(6)
P(3)-O(1)	1.463(4)	C(22)-C(23)	1.388(6)
P(3)-O(2)	1.474(4)	C(22)-H(22A)	0.9500
P(3)-O(4)	1.628(3)	C(23)-C(24)	1.380(6)
P(3)-O(3)	1.632(4)	C(23)-H(23A)	0.9500
O(3)-C(27)	1.384(6)	C(24)-C(25)	1.387(6)
O(4)-C(33)	1.393(5)	C(24)-H(24A)	0.9500
C(1)-C(6)	1.393(6)	C(25)-C(26)	1.375(6)
C(1)-C(2)	1.400(6)	C(25)-H(25A)	0.9500
C(2)-C(3)	1.394(6)	C(26)-H(26A)	0.9500
C(2)-H(2B)	0.9500	C(27)-C(28)	1.387(7)
C(3)-C(4)	1.375(7)	C(27)-C(32)	1.400(7)
C(3)-H(3A)	0.9500	C(28)-C(29)	1.393(8)
C(4)-C(5)	1.382(8)	C(28)-H(28A)	0.9500
C(4)-H(4A)	0.9500	C(29)-C(30)	1.387(9)
C(5)-C(6)	1.388(6)	C(29)-H(29A)	0.9500
C(5)-H(5A)	0.9500	C(30)-C(31)	1.393(9)
C(6)-H(6A)	0.9500	C(30)-H(30A)	0.9500
C(7)-C(8)	1.384(6)	C(31)-C(32)	1.381(8)
C(7)-C(12)	1.398(6)	C(31)-H(31A)	0.9500
C(8)-C(9)	1.393(6)	C(32)-H(32A)	0.9500
C(8)-H(8A)	0.9500	C(33)-C(38)	1.386(7)
C(9)-C(10)	1.387(7)	C(33)-C(34)	1.388(7)
C(9)-H(9A)	0.9500	C(34)-C(35)	1.390(7)
C(10)-C(11)	1.379(7)	C(34)-H(34A)	0.9500
C(10)-H(10A)	0.9500	C(35)-C(36)	1.378(7)
C(11)-C(12)	1.391(6)	C(35)-H(35A)	0.9500
C(11)-H(11A)	0.9500	C(36)-C(37)	1.372(8)
C(12)-H(12A)	0.9500	C(36)-H(36A)	0.9500
C(13)-C(14)	1.523(6)	C(37)-C(38)	1.376(7)
C(13)-H(13A)	0.9900	C(37)-H(37A)	0.9500
C(13)-H(13B)	0.9900	C(38)-H(38A)	0.9500
C(14)-H(14A)	0.9900	C(39)-H(39A)	0.9900
C(14)-H(14B)	0.9900	C(39)-H(39B)	0.9900
C(15)-C(16)	1.389(6)		

P(2)#1-Pd-P(2)	180.00(6)	C(1)-P(1)-C(13)	106.2(2)
P(2)#1-Pd-P(1)	97.34(4)	C(7)-P(1)-C(13)	104.9(2)
P(2)-Pd-P(1)	82.66(4)	C(1)-P(1)-Pd	124.65(14)
P(2)#1-Pd-P(1)#1	82.66(4)	C(7)-P(1)-Pd	111.70(14)
P(2)-Pd-P(1)#1	97.34(4)	C(13)-P(1)-Pd	104.03(14)
P(1)-Pd-P(1)#1	180.00(7)	C(15)-P(2)-C(21)	107.01(19)
C(1)-P(1)-C(7)	103.79(19)	C(15)-P(2)-C(14)	103.86(19)

C(21)-P(2)-C(14)	105.90(19)	P(2)-C(14)-H(14A)	109.4
C(15)-P(2)-Pd	119.01(14)	C(13)-C(14)-H(14B)	109.4
C(21)-P(2)-Pd	111.31(13)	P(2)-C(14)-H(14B)	109.4
C(14)-P(2)-Pd	108.79(13)	H(14A)-C(14)-H(14B)	108.0
O(1)-P(3)-O(2)	121.5(2)	C(16)-C(15)-C(20)	119.9(4)
O(1)-P(3)-O(4)	111.00(19)	C(16)-C(15)-P(2)	121.0(3)
O(2)-P(3)-O(4)	104.85(19)	C(20)-C(15)-P(2)	119.1(3)
O(1)-P(3)-O(3)	106.1(2)	C(17)-C(16)-C(15)	119.6(4)
O(2)-P(3)-O(3)	109.34(19)	C(17)-C(16)-H(16A)	120.2
O(4)-P(3)-O(3)	102.44(18)	C(15)-C(16)-H(16A)	120.2
C(27)-O(3)-P(3)	122.1(3)	C(18)-C(17)-C(16)	120.2(4)
C(33)-O(4)-P(3)	124.8(3)	C(18)-C(17)-H(17A)	119.9
C(6)-C(1)-C(2)	119.4(4)	C(16)-C(17)-H(17A)	119.9
C(6)-C(1)-P(1)	120.3(3)	C(19)-C(18)-C(17)	120.3(4)
C(2)-C(1)-P(1)	120.2(3)	C(19)-C(18)-H(18A)	119.8
C(3)-C(2)-C(1)	119.7(4)	C(17)-C(18)-H(18A)	119.8
C(3)-C(2)-H(2B)	120.1	C(18)-C(19)-C(20)	119.9(4)
C(1)-C(2)-H(2B)	120.1	C(18)-C(19)-H(19A)	120.0
C(4)-C(3)-C(2)	120.0(5)	C(20)-C(19)-H(19A)	120.0
C(4)-C(3)-H(3A)	120.0	C(19)-C(20)-C(15)	120.1(4)
C(2)-C(3)-H(3A)	120.0	C(19)-C(20)-H(20A)	120.0
C(3)-C(4)-C(5)	120.8(4)	C(15)-C(20)-H(20A)	120.0
C(3)-C(4)-H(4A)	119.6	C(22)-C(21)-C(26)	119.3(4)
C(5)-C(4)-H(4A)	119.6	C(22)-C(21)-P(2)	123.7(3)
C(4)-C(5)-C(6)	119.7(5)	C(26)-C(21)-P(2)	117.1(3)
C(4)-C(5)-H(5A)	120.1	C(21)-C(22)-C(23)	120.2(4)
C(6)-C(5)-H(5A)	120.1	C(21)-C(22)-H(22A)	119.9
C(5)-C(6)-C(1)	120.2(4)	C(23)-C(22)-H(22A)	119.9
C(5)-C(6)-H(6A)	119.9	C(24)-C(23)-C(22)	120.2(4)
C(1)-C(6)-H(6A)	119.9	C(24)-C(23)-H(23A)	119.9
C(8)-C(7)-C(12)	119.9(4)	C(22)-C(23)-H(23A)	119.9
C(8)-C(7)-P(1)	121.8(3)	C(23)-C(24)-C(25)	119.6(4)
C(12)-C(7)-P(1)	118.3(3)	C(23)-C(24)-H(24A)	120.2
C(7)-C(8)-C(9)	119.9(4)	C(25)-C(24)-H(24A)	120.2
C(7)-C(8)-H(8A)	120.0	C(26)-C(25)-C(24)	120.6(4)
C(9)-C(8)-H(8A)	120.0	C(26)-C(25)-H(25A)	119.7
C(10)-C(9)-C(8)	119.9(4)	C(24)-C(25)-H(25A)	119.7
C(10)-C(9)-H(9A)	120.0	C(25)-C(26)-C(21)	120.1(4)
C(8)-C(9)-H(9A)	120.0	C(25)-C(26)-H(26A)	120.0
C(11)-C(10)-C(9)	120.4(4)	C(21)-C(26)-H(26A)	120.0
C(11)-C(10)-H(10A)	119.8	O(3)-C(27)-C(28)	116.7(5)
C(9)-C(10)-H(10A)	119.8	O(3)-C(27)-C(32)	122.7(5)
C(10)-C(11)-C(12)	120.0(4)	C(28)-C(27)-C(32)	120.6(5)
C(10)-C(11)-H(11A)	120.0	C(27)-C(28)-C(29)	120.2(6)
C(12)-C(11)-H(11A)	120.0	C(27)-C(28)-H(28A)	119.9
C(11)-C(12)-C(7)	119.8(4)	C(29)-C(28)-H(28A)	119.9
C(11)-C(12)-H(12A)	120.1	C(30)-C(29)-C(28)	119.2(6)
C(7)-C(12)-H(12A)	120.1	C(30)-C(29)-H(29A)	120.4
C(14)-C(13)-P(1)	107.9(3)	C(28)-C(29)-H(29A)	120.4
C(14)-C(13)-H(13A)	110.1	C(29)-C(30)-C(31)	120.5(6)
P(1)-C(13)-H(13A)	110.1	C(29)-C(30)-H(30A)	119.8
C(14)-C(13)-H(13B)	110.1	C(31)-C(30)-H(30A)	119.8
P(1)-C(13)-H(13B)	110.1	C(32)-C(31)-C(30)	120.6(6)
H(13A)-C(13)-H(13B)	108.4	C(32)-C(31)-H(31A)	119.7
C(13)-C(14)-P(2)	111.1(3)	C(30)-C(31)-H(31A)	119.7
C(13)-C(14)-H(14A)	109.4	C(31)-C(32)-C(27)	118.9(6)

C(31)-C(32)-H(32A)	120.5	C(35)-C(36)-H(36A)	120.3
C(27)-C(32)-H(32A)	120.5	C(36)-C(37)-C(38)	120.8(5)
C(38)-C(33)-C(34)	121.0(4)	C(36)-C(37)-H(37A)	119.6
C(38)-C(33)-O(4)	115.5(4)	C(38)-C(37)-H(37A)	119.6
C(34)-C(33)-O(4)	123.5(4)	C(37)-C(38)-C(33)	119.5(5)
C(33)-C(34)-C(35)	117.8(5)	C(37)-C(38)-H(38A)	120.3
C(33)-C(34)-H(34A)	121.1	C(33)-C(38)-H(38A)	120.3
C(35)-C(34)-H(34A)	121.1	Cl(1)-C(39)-Cl(2)	112.2(3)
C(36)-C(35)-C(34)	121.5(5)	Cl(1)-C(39)-H(39A)	109.2
C(36)-C(35)-H(35A)	119.2	Cl(2)-C(39)-H(39A)	109.2
C(34)-C(35)-H(35A)	119.2	Cl(1)-C(39)-H(39B)	109.2
C(37)-C(36)-C(35)	119.3(5)	Cl(2)-C(39)-H(39B)	109.2
C(37)-C(36)-H(36A)	120.3	H(39A)-C(39)-H(39B)	107.9

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

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for compound **9**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Pd	12(1)	10(1)	11(1)	-1(1)	1(1)	1(1)
Cl(1)	77(1)	50(1)	29(1)	-8(1)	2(1)	17(1)
Cl(2)	63(1)	39(1)	33(1)	-1(1)	17(1)	2(1)
P(1)	15(1)	11(1)	14(1)	-1(1)	2(1)	2(1)
P(2)	17(1)	12(1)	15(1)	0(1)	3(1)	0(1)
P(3)	30(1)	26(1)	20(1)	1(1)	6(1)	0(1)
O(1)	35(2)	33(2)	29(2)	-8(2)	13(2)	-4(2)
O(2)	29(2)	31(2)	33(2)	-1(2)	8(2)	5(2)
O(3)	35(2)	26(2)	34(2)	3(2)	4(2)	0(2)
O(4)	35(2)	28(2)	25(2)	3(1)	11(2)	6(2)
C(1)	19(2)	19(2)	12(2)	-1(2)	0(2)	7(2)
C(2)	23(2)	27(3)	19(2)	-2(2)	5(2)	1(2)
C(3)	31(3)	35(3)	21(2)	1(2)	10(2)	2(2)
C(4)	38(3)	45(3)	17(2)	-4(2)	7(2)	12(3)
C(5)	43(3)	29(3)	16(2)	-8(2)	-2(2)	6(2)
C(6)	25(2)	18(2)	20(2)	1(2)	1(2)	4(2)
C(7)	21(2)	13(2)	12(2)	-1(2)	-2(2)	-1(2)
C(8)	23(2)	17(2)	20(2)	-2(2)	2(2)	0(2)
C(9)	26(3)	30(3)	20(2)	-1(2)	5(2)	-6(2)
C(10)	41(3)	17(2)	24(2)	4(2)	1(2)	-7(2)
C(11)	33(3)	14(2)	23(2)	-2(2)	0(2)	5(2)
C(12)	21(2)	19(2)	18(2)	-5(2)	1(2)	1(2)
C(13)	14(2)	17(2)	20(2)	-3(2)	2(2)	3(2)
C(14)	16(2)	12(2)	18(2)	-3(2)	2(2)	2(2)
C(15)	25(2)	10(2)	15(2)	-1(2)	5(2)	-4(2)
C(16)	23(2)	15(2)	22(2)	2(2)	4(2)	2(2)
C(17)	25(3)	24(2)	36(3)	4(2)	12(2)	-3(2)
C(18)	36(3)	27(3)	27(3)	7(2)	19(2)	-1(2)
C(19)	40(3)	24(2)	20(2)	7(2)	6(2)	1(2)
C(20)	21(2)	20(2)	20(2)	2(2)	2(2)	0(2)
C(21)	13(2)	10(2)	16(2)	-1(2)	-2(2)	-1(2)
C(22)	23(2)	16(2)	15(2)	2(2)	5(2)	0(2)
C(23)	29(3)	21(2)	18(2)	-7(2)	2(2)	4(2)
C(24)	24(2)	15(2)	31(3)	-2(2)	0(2)	-5(2)
C(25)	28(3)	19(2)	23(2)	2(2)	9(2)	-3(2)
C(26)	23(2)	17(2)	18(2)	-5(2)	4(2)	1(2)
C(27)	23(3)	22(2)	46(3)	2(2)	10(2)	7(2)
C(28)	32(3)	39(3)	41(3)	-5(3)	8(2)	-2(2)
C(29)	33(3)	45(4)	64(4)	-12(3)	11(3)	-1(3)
C(30)	28(3)	27(3)	94(5)	-12(3)	15(3)	-1(2)
C(31)	28(3)	34(3)	80(5)	13(3)	15(3)	0(2)
C(32)	35(3)	29(3)	51(3)	7(3)	13(3)	3(2)
C(33)	22(2)	24(2)	33(3)	-6(2)	8(2)	3(2)
C(34)	23(2)	28(3)	33(3)	-1(2)	6(2)	-1(2)
C(35)	19(2)	31(3)	48(3)	4(2)	7(2)	2(2)
C(36)	18(2)	35(3)	51(3)	-11(3)	5(2)	4(2)
C(37)	35(3)	49(4)	31(3)	-5(3)	3(2)	10(3)
C(38)	37(3)	41(3)	30(3)	4(2)	3(2)	9(2)
C(39)	43(3)	40(3)	29(3)	-2(2)	0(2)	-12(3)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for compound **9**.

	x	y	z	U(eq)
H(2B)	2278	546	1139	28
H(3A)	1848	911	2340	34
H(4A)	2817	1919	3059	39
H(5A)	4257	2550	2619	36
H(6A)	4709	2190	1427	26
H(8A)	5709	1626	-439	24
H(9A)	6228	2874	-887	30
H(10A)	5125	4000	-850	33
H(11A)	3498	3889	-386	29
H(12A)	2975	2647	67	23
H(13A)	2267	342	-203	21
H(13B)	2066	1287	-364	21
H(14A)	2948	1257	-1422	19
H(14B)	2150	497	-1525	19
H(16A)	6116	511	-1447	24
H(17A)	6856	1012	-2488	33
H(18A)	5759	1228	-3671	34
H(19A)	3919	949	-3824	33
H(20A)	3164	465	-2788	25
H(22A)	3826	-1022	-2579	21
H(23A)	2977	-2255	-2905	27
H(24A)	2084	-2921	-2027	29
H(25A)	2023	-2344	-824	27
H(26A)	2831	-1106	-503	23
H(28A)	10570	2150	1084	45
H(29A)	11499	3375	1322	56
H(30A)	11701	4215	287	59
H(31A)	10988	3838	-971	56
H(32A)	10030	2635	-1210	45
H(34A)	9748	96	-786	33
H(35A)	10647	-981	-1268	39
H(36A)	10948	-987	-2535	42
H(37A)	10406	107	-3327	46
H(38A)	9489	1177	-2878	43
H(39A)	8181	1665	-5071	46
H(39B)	9072	967	-4951	46

Table 6. Torsion angles [°] for compound **9**.

P(2)#1-Pd-P(1)-C(1)	-26.14(18)	C(15)-P(2)-C(14)-C(13)	-147.6(3)
P(2)-Pd-P(1)-C(1)	153.86(18)	C(21)-P(2)-C(14)-C(13)	99.9(3)
P(1)#1-Pd-P(1)-C(1)	145(68)	Pd-P(2)-C(14)-C(13)	-19.9(3)
P(2)#1-Pd-P(1)-C(7)	99.80(14)	C(21)-P(2)-C(15)-C(16)	-119.4(4)
P(2)-Pd-P(1)-C(7)	-80.20(14)	C(14)-P(2)-C(15)-C(16)	128.9(4)
P(1)#1-Pd-P(1)-C(7)	-89(68)	Pd-P(2)-C(15)-C(16)	7.8(4)
P(2)#1-Pd-P(1)-C(13)	-147.62(15)	C(21)-P(2)-C(15)-C(20)	62.2(4)
P(2)-Pd-P(1)-C(13)	32.38(15)	C(14)-P(2)-C(15)-C(20)	-49.5(4)
P(1)#1-Pd-P(1)-C(13)	23(68)	Pd-P(2)-C(15)-C(20)	-170.6(3)
P(2)#1-Pd-P(2)-C(15)	-69(100)	C(20)-C(15)-C(16)-C(17)	-1.0(6)
P(1)-Pd-P(2)-C(15)	108.44(16)	P(2)-C(15)-C(16)-C(17)	-179.3(3)
P(1)#1-Pd-P(2)-C(15)	-71.56(16)	C(15)-C(16)-C(17)-C(18)	0.2(7)
P(2)#1-Pd-P(2)-C(21)	56(100)	C(16)-C(17)-C(18)-C(19)	0.2(7)
P(1)-Pd-P(2)-C(21)	-126.44(15)	C(17)-C(18)-C(19)-C(20)	0.2(7)
P(1)#1-Pd-P(2)-C(21)	53.56(15)	C(18)-C(19)-C(20)-C(15)	-1.0(7)
P(2)#1-Pd-P(2)-C(14)	172(100)	C(16)-C(15)-C(20)-C(19)	1.4(6)
P(1)-Pd-P(2)-C(14)	-10.12(15)	P(2)-C(15)-C(20)-C(19)	179.8(4)
P(1)#1-Pd-P(2)-C(14)	169.88(15)	C(15)-P(2)-C(21)-C(22)	0.2(4)
O(1)-P(3)-O(3)-C(27)	-164.2(3)	C(14)-P(2)-C(21)-C(22)	110.5(4)
O(2)-P(3)-O(3)-C(27)	-31.5(4)	Pd-P(2)-C(21)-C(22)	-131.4(3)
O(4)-P(3)-O(3)-C(27)	79.3(4)	C(15)-P(2)-C(21)-C(26)	-179.0(3)
O(1)-P(3)-O(4)-C(33)	-45.9(4)	C(14)-P(2)-C(21)-C(26)	-68.7(4)
O(2)-P(3)-O(4)-C(33)	-178.8(4)	Pd-P(2)-C(21)-C(26)	49.4(3)
O(3)-P(3)-O(4)-C(33)	67.0(4)	C(26)-C(21)-C(22)-C(23)	1.9(6)
C(7)-P(1)-C(1)-C(6)	-33.4(4)	P(2)-C(21)-C(22)-C(23)	-177.3(3)
C(13)-P(1)-C(1)-C(6)	-143.7(3)	C(21)-C(22)-C(23)-C(24)	-1.5(7)
Pd-P(1)-C(1)-C(6)	95.8(4)	C(22)-C(23)-C(24)-C(25)	0.2(7)
C(7)-P(1)-C(1)-C(2)	144.3(4)	C(23)-C(24)-C(25)-C(26)	0.5(7)
C(13)-P(1)-C(1)-C(2)	34.0(4)	C(24)-C(25)-C(26)-C(21)	0.0(7)
Pd-P(1)-C(1)-C(2)	-86.5(4)	C(22)-C(21)-C(26)-C(25)	-1.2(6)
C(6)-C(1)-C(2)-C(3)	-0.6(7)	P(2)-C(21)-C(26)-C(25)	178.1(3)
P(1)-C(1)-C(2)-C(3)	-178.4(4)	P(3)-O(3)-C(27)-C(28)	139.6(4)
C(1)-C(2)-C(3)-C(4)	0.9(7)	P(3)-O(3)-C(27)-C(32)	-42.0(6)
C(2)-C(3)-C(4)-C(5)	-1.0(8)	O(3)-C(27)-C(28)-C(29)	-179.1(5)
C(3)-C(4)-C(5)-C(6)	0.7(8)	C(32)-C(27)-C(28)-C(29)	2.4(8)
C(4)-C(5)-C(6)-C(1)	-0.4(7)	C(27)-C(28)-C(29)-C(30)	-1.5(8)
C(2)-C(1)-C(6)-C(5)	0.4(7)	C(28)-C(29)-C(30)-C(31)	-0.2(8)
P(1)-C(1)-C(6)-C(5)	178.1(3)	C(29)-C(30)-C(31)-C(32)	1.0(9)
C(1)-P(1)-C(7)-C(8)	129.4(3)	C(30)-C(31)-C(32)-C(27)	-0.1(8)
C(13)-P(1)-C(7)-C(8)	-119.3(4)	O(3)-C(27)-C(32)-C(31)	180.0(5)
Pd-P(1)-C(7)-C(8)	-7.3(4)	C(28)-C(27)-C(32)-C(31)	-1.6(8)
C(1)-P(1)-C(7)-C(12)	-50.7(4)	P(3)-O(4)-C(33)-C(38)	176.2(4)
C(13)-P(1)-C(7)-C(12)	60.5(4)	P(3)-O(4)-C(33)-C(34)	-4.8(7)
Pd-P(1)-C(7)-C(12)	172.6(3)	C(38)-C(33)-C(34)-C(35)	-3.2(7)
C(12)-C(7)-C(8)-C(9)	0.9(6)	O(4)-C(33)-C(34)-C(35)	177.7(4)
P(1)-C(7)-C(8)-C(9)	-179.3(3)	C(33)-C(34)-C(35)-C(36)	1.2(7)
C(7)-C(8)-C(9)-C(10)	-0.3(7)	C(34)-C(35)-C(36)-C(37)	1.3(8)
C(8)-C(9)-C(10)-C(11)	-0.5(7)	C(35)-C(36)-C(37)-C(38)	-1.8(8)
C(9)-C(10)-C(11)-C(12)	0.6(7)	C(36)-C(37)-C(38)-C(33)	-0.2(8)
C(10)-C(11)-C(12)-C(7)	0.0(7)	C(34)-C(33)-C(38)-C(37)	2.8(8)
C(8)-C(7)-C(12)-C(11)	-0.7(6)	O(4)-C(33)-C(38)-C(37)	-178.1(5)
P(1)-C(7)-C(12)-C(11)	179.4(3)		
C(1)-P(1)-C(13)-C(14)	172.5(3)		
C(7)-P(1)-C(13)-C(14)	63.0(3)		
Pd-P(1)-C(13)-C(14)	-54.4(3)		
P(1)-C(13)-C(14)-P(2)	47.9(3)		

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

Crystallographic Experimental Section for Compound 13

Data Collection

A colorless crystal with approximate dimensions 0.38 x 0.26 x 0.18 mm^3 was selected under oil under ambient conditions and attached to the tip of a glass capillary. The crystal was mounted in a stream of cold nitrogen at 173(2) K and centered in the X-ray beam by using a video camera.

The crystal evaluation and data collection were performed on a Bruker CCD-1000 diffractometer with Mo K_α (λ = 0.71073 Å) radiation and the diffractometer to crystal distance of 4.9 cm.

The initial cell constants were obtained from three series of ω scans at different starting angles. Each series consisted of 20 frames collected at intervals of 0.3º in a 6º range about ω with the exposure time of 10 seconds per frame. A total of 189 reflections was obtained. The reflections were successfully indexed by an automated indexing routine built in the SMART program. The final cell constants were calculated from a set of 18188 strong reflections from the actual data collection.

The data were collected by using the hemisphere data collection routine. The reciprocal space was surveyed to the extent of a full sphere to a resolution of 0.80 Å. A total of 47497 data were harvested by collecting three sets of frames with 0.3º scans in ω with an exposure time 30 sec per frame. These highly redundant datasets were corrected for Lorentz and polarization effects. The absorption correction was performed by using program *DIFABS*.[2]

Structure Solution and Refinement

The systematic absences in the diffraction data were uniquely consistent for the space group $P2_1/n$ that yielded chemically reasonable and computationally stable results of refinement.[1]

A successful solution by the direct methods provided most non-hydrogen atoms from the *E*-map. The remaining non-hydrogen atoms were located in an alternating series of least-squares cycles and difference Fourier maps. All non-hydrogen atoms were refined with anisotropic displacement coefficients. All hydrogen atoms were included in the structure factor calculation

at idealized positions and were allowed to ride on the neighboring atoms with relative isotropic displacement coefficients. There are two symmetry independent complexes with similar geometries in the lattice. The biggest difference is in the tilt angle of one phenyl ring, Figure 3.

The final least-squares refinement of 811 parameters against 12541 data resulted in residuals R (based on F^2 for $I \geq 2\sigma$) and wR (based on F^2 for all data) of 0.0322 and 0.0728, respectively. The final difference Fourier map was featureless.

The ORTEP diagrams are drawn with 50% probability ellipsoids.

Table 1. Crystal data and structure refinement for compound **13**.

Identification code	sto02	
Empirical formula	$C_{34}\ H_{28}\ N_2\ O_6\ P_2\ Pd$	
Formula weight	728.92	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 9.3946(3) Å	α= 90°.
	b = 34.3472(9) Å	β= 101.726(1)°
	c = 19.4549(5) Å	γ = 90°.
Volume	6146.7(3) $Å^3$	
Z	8	
Density (calculated)	1.575 Mg/m^3	
Absorption coefficient	0.758 mm^{-1}	
F(000)	2960	
Crystal size	0.38 x 0.26 x 0.18 mm^3	
Theta range for data collection	1.22 to 26.36°.	
Index ranges	-11<=h<=11, 0<=k<=42, 0<=l<=24	
Reflections collected	47497	
Independent reflections	12541 [R(int) = 0.0337	
Completeness to theta = 26.36°	99.8 %	
Absorption correction	Empirical with DIFABS	
Max. and min. transmission	0.8757 and 0.7616	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	12541 / 0 / 811	
Goodness-of-fit on F^2	1.063	
Final R indices [I>2sigma(I)]	R1 = 0.0322, wR2 = 0.0700	
R indices (all data)	R1 = 0.0395, wR2 = 0.0728	
Largest diff. peak and hole	0.401 and -0.440 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for compound **13**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Pd(1)	8908(1)	8276(1)	7605(1)	24(1)
Pd(2)	9656(1)	10178(1)	2493(1)	23(1)
P(1)	10297(1)	7862(1)	8339(1)	23(1)
P(2)	8918(1)	7870(1)	6701(1)	25(1)
P(1A)	10930(1)	9778(1)	3297(1)	24(1)
P(2A)	9520(1)	9725(1)	1646(1)	24(1)
O(1)	11787(2)	7996(1)	8636(1)	30(1)
O(2)	10280(2)	7419(1)	8062(1)	25(1)
O(3)	9472(2)	7819(1)	8997(1)	27(1)
O(4)	8459(2)	8055(1)	6002(1)	34(1)
O(5)	10443(2)	7640(1)	6743(1)	29(1)
O(6)	7931(2)	7498(1)	6800(1)	28(1)
O(1A)	12447(2)	9898(1)	3576(1)	31(1)
O(2A)	10838(2)	9315(1)	3134(1)	26(1)
O(3A)	10106(2)	9808(1)	3961(1)	27(1)
O(4A)	8953(2)	9856(1)	913(1)	35(1)
O(5A)	11058(2)	9499(1)	1695(1)	27(1)
O(6A)	8589(2)	9370(1)	1864(1)	27(1)
N(1)	8951(2)	8711(1)	8415(1)	27(1)
N(2)	7454(2)	8721(1)	7084(1)	27(1)
N(1A)	9813(2)	10637(1)	3256(1)	27(1)
N(2A)	8301(2)	10623(1)	1930(1)	28(1)
C(1)	9751(3)	8702(1)	9071(1)	35(1)
C(2)	9588(3)	8968(1)	9583(2)	39(1)
C(3)	8586(3)	9260(1)	9417(2)	43(1)
C(4)	7782(3)	9281(1)	8733(2)	41(1)
C(5)	7988(3)	9005(1)	8244(1)	30(1)
C(6)	7189(3)	9017(1)	7501(1)	31(1)
C(7)	6259(3)	9319(1)	7241(2)	43(1)
C(8)	5569(4)	9323(1)	6544(2)	49(1)
C(9)	5823(3)	9020(1)	6123(2)	43(1)
C(10)	6764(3)	8725(1)	6406(1)	35(1)
C(11)	11311(2)	7141(1)	8351(1)	24(1)
C(12)	11859(3)	6910(1)	7882(1)	30(1)
C(13)	12833(3)	6617(1)	8141(2)	40(1)
C(14)	13255(3)	6559(1)	8861(2)	41(1)
C(15)	12698(3)	6792(1)	9321(1)	34(1)
C(16)	11716(3)	7084(1)	9070(1)	27(1)
C(17)	7980(3)	7742(1)	8903(1)	32(1)
C(18)	7333(4)	7441(1)	8504(2)	61(1)
C(19)	5851(4)	7377(2)	8461(2)	92(2)
C(20)	5076(4)	7599(2)	8823(2)	89(2)
C(21)	5748(5)	7896(1)	9232(3)	88(2)
C(22)	7206(4)	7974(1)	9268(2)	64(1)
C(23)	11629(3)	7747(1)	6465(1)	27(1)
C(24)	11526(3)	7778(1)	5746(1)	32(1)
C(25)	12788(4)	7847(1)	5495(2)	42(1)
C(26)	14108(4)	7885(1)	5953(2)	49(1)
C(27)	14194(3)	7856(1)	6668(2)	45(1)
C(28)	12950(3)	7789(1)	6929(1)	33(1)
C(29)	7967(3)	7153(1)	6413(1)	28(1)

C(30)	7701(3)	7154(1)	5689(1)	36(1)
C(31)	7713(3)	6804(1)	5335(1)	40(1)
C(32)	7960(3)	6457(1)	5704(2)	41(1)
C(33)	8239(3)	6462(1)	6430(2)	40(1)
C(34)	8240(3)	6811(1)	6790(1)	34(1)
C(1A)	10703(3)	10651(1)	3890(1)	32(1)
C(2A)	10635(3)	10943(1)	4371(2)	35(1)
C(3A)	9630(3)	11235(1)	4196(2)	38(1)
C(4A)	8729(3)	11229(1)	3538(2)	38(1)
C(5A)	8848(3)	10930(1)	3071(1)	30(1)
C(6A)	7990(3)	10918(1)	2341(1)	32(1)
C(7A)	6960(4)	11199(1)	2081(2)	47(1)
C(8A)	6225(4)	11175(1)	1388(2)	56(1)
C(9A)	6550(4)	10880(1)	971(2)	47(1)
C(10A)	7598(3)	10612(1)	1258(1)	36(1)
C(11A)	11852(3)	9070(1)	3557(1)	25(1)
C(12A)	12855(3)	8882(1)	3245(1)	31(1)
C(13A)	13862(3)	8637(1)	3646(1)	34(1)
C(14A)	13865(3)	8577(1)	4344(2)	36(1)
C(15A)	12851(3)	8767(1)	4650(1)	37(1)
C(16A)	11841(3)	9014(1)	4264(1)	31(1)
C(17A)	8642(3)	9742(1)	3956(1)	27(1)
C(18A)	7792(3)	9488(1)	3499(1)	33(1)
C(19A)	6373(3)	9420(1)	3570(2)	41(1)
C(20A)	5810(3)	9601(1)	4089(2)	45(1)
C(21A)	6671(3)	9857(1)	4544(2)	44(1)
C(22A)	8085(3)	9931(1)	4474(1)	36(1)
C(23A)	12272(3)	9644(1)	1472(1)	26(1)
C(24A)	13547(3)	9676(1)	1975(1)	29(1)
C(25A)	14824(3)	9779(1)	1768(2)	36(1)
C(26A)	14814(3)	9859(1)	1067(2)	41(1)
C(27A)	13522(3)	9838(1)	576(2)	40(1)
C(28A)	12240(3)	9728(1)	774(1)	31(1)
C(29A)	8417(3)	9000(1)	1548(1)	27(1)
C(30A)	7059(3)	8894(1)	1204(2)	58(1)
C(31A)	6836(4)	8516(1)	954(3)	85(2)
C(32A)	7954(4)	8253(1)	1050(2)	59(1)
C(33A)	9296(3)	8364(1)	1391(2)	46(1)
C(34A)	9539(3)	8737(1)	1654(2)	37(1)

Table 3. Bond lengths [Å] and angles [°] for compound **13**.

Pd(1)-N(2)	2.160(2)	C(17)-C(18)	1.359(4)
Pd(1)-N(1)	2.165(2)	C(17)-C(22)	1.372(4)
Pd(1)-P(1)	2.2377(6)	C(18)-C(19)	1.395(5)
Pd(1)-P(2)	2.2466(7)	C(19)-C(20)	1.348(7)
Pd(2)-N(2A)	2.144(2)	C(20)-C(21)	1.367(7)
Pd(2)-N(1A)	2.151(2)	C(21)-C(22)	1.384(5)
Pd(2)-P(1A)	2.2369(6)	C(23)-C(28)	1.386(4)
Pd(2)-P(2A)	2.2509(6)	C(23)-C(24)	1.386(3)
P(1)-O(1)	1.4756(18)	C(24)-C(25)	1.391(4)
P(1)-O(2)	1.6106(17)	C(25)-C(26)	1.379(5)
P(1)-O(3)	1.6327(17)	C(26)-C(27)	1.380(4)
P(2)-O(4)	1.4827(18)	C(27)-C(28)	1.384(4)
P(2)-O(6)	1.6140(18)	C(29)-C(34)	1.379(4)
P(2)-O(5)	1.6218(18)	C(29)-C(30)	1.380(3)
P(1A)-O(1A)	1.4761(18)	C(30)-C(31)	1.384(4)
P(1A)-O(2A)	1.6201(17)	C(31)-C(32)	1.386(4)
P(1A)-O(3A)	1.6387(17)	C(32)-C(33)	1.383(4)
P(2A)-O(4A)	1.4859(18)	C(33)-C(34)	1.390(4)
P(2A)-O(6A)	1.6064(18)	C(1A)-C(2A)	1.385(4)
P(2A)-O(5A)	1.6248(17)	C(2A)-C(3A)	1.371(4)
O(2)-C(11)	1.395(3)	C(3A)-C(4A)	1.382(4)
O(3)-C(17)	1.402(3)	C(4A)-C(5A)	1.390(4)
O(5)-C(23)	1.383(3)	C(5A)-C(6A)	1.485(4)
O(6)-C(29)	1.407(3)	C(6A)-C(7A)	1.386(4)
O(2A)-C(11A)	1.405(3)	C(7A)-C(8A)	1.385(4)
O(3A)-C(17A)	1.391(3)	C(8A)-C(9A)	1.372(5)
O(5A)-C(23A)	1.392(3)	C(9A)-C(10A)	1.381(4)
O(6A)-C(29A)	1.407(3)	C(11A)-C(12A)	1.381(3)
N(1)-C(1)	1.344(3)	C(11A)-C(16A)	1.392(3)
N(1)-C(5)	1.353(3)	C(12A)-C(13A)	1.384(4)
N(2)-C(10)	1.345(3)	C(13A)-C(14A)	1.372(4)
N(2)-C(6)	1.356(3)	C(14A)-C(15A)	1.384(4)
N(1A)-C(1A)	1.343(3)	C(15A)-C(16A)	1.377(4)
N(1A)-C(5A)	1.353(3)	C(17A)-C(18A)	1.379(4)
N(2A)-C(10A)	1.341(3)	C(17A)-C(22A)	1.388(4)
N(2A)-C(6A)	1.358(3)	C(18A)-C(19A)	1.388(4)
C(1)-C(2)	1.381(4)	C(19A)-C(20A)	1.381(4)
C(2)-C(3)	1.370(4)	C(20A)-C(21A)	1.385(4)
C(3)-C(4)	1.391(4)	C(21A)-C(22A)	1.386(4)
C(4)-C(5)	1.384(4)	C(23A)-C(28A)	1.383(3)
C(5)-C(6)	1.487(4)	C(23A)-C(24A)	1.389(4)
C(6)-C(7)	1.385(4)	C(24A)-C(25A)	1.387(4)
C(7)-C(8)	1.379(4)	C(25A)-C(26A)	1.389(4)
C(8)-C(9)	1.374(4)	C(26A)-C(27A)	1.385(4)
C(9)-C(10)	1.384(4)	C(27A)-C(28A)	1.389(4)
C(11)-C(12)	1.385(3)	C(29A)-C(30A)	1.364(4)
C(11)-C(16)	1.387(3)	C(29A)-C(34A)	1.372(4)
C(12)-C(13)	1.383(4)	C(30A)-C(31A)	1.389(4)
C(13)-C(14)	1.390(4)	C(31A)-C(32A)	1.369(5)
C(14)-C(15)	1.382(4)	C(32A)-C(33A)	1.354(4)
C(15)-C(16)	1.383(4)	C(33A)-C(34A)	1.381(4)
N(2)-Pd(1)-N(1)	76.70(8)	N(1)-Pd(1)-P(1)	92.97(6)
N(2)-Pd(1)-P(1)	168.62(6)	N(2)-Pd(1)-P(2)	100.05(6)

N(1)-Pd(1)-P(2)	174.57(6)	N(1)-C(5)-C(4)	121.5(2)
P(1)-Pd(1)-P(2)	90.60(2)	N(1)-C(5)-C(6)	116.0(2)
N(2A)-Pd(2)-N(1A)	77.23(8)	C(4)-C(5)-C(6)	122.5(2)
N(2A)-Pd(2)-P(1A)	166.68(6)	N(2)-C(6)-C(7)	121.5(2)
N(1A)-Pd(2)-P(1A)	90.88(6)	N(2)-C(6)-C(5)	116.4(2)
N(2A)-Pd(2)-P(2A)	100.36(6)	C(7)-C(6)-C(5)	122.1(2)
N(1A)-Pd(2)-P(2A)	176.43(6)	C(8)-C(7)-C(6)	120.1(3)
P(1A)-Pd(2)-P(2A)	91.78(2)	C(9)-C(8)-C(7)	118.4(3)
O(1)-P(1)-O(2)	111.71(10)	C(8)-C(9)-C(10)	119.4(3)
O(1)-P(1)-O(3)	106.54(10)	N(2)-C(10)-C(9)	122.7(3)
O(2)-P(1)-O(3)	101.80(9)	C(12)-C(11)-C(16)	121.4(2)
O(1)-P(1)-Pd(1)	115.78(8)	C(12)-C(11)-O(2)	116.7(2)
O(2)-P(1)-Pd(1)	114.90(6)	C(16)-C(11)-O(2)	121.9(2)
O(3)-P(1)-Pd(1)	104.40(6)	C(13)-C(12)-C(11)	119.0(2)
O(4)-P(2)-O(6)	112.44(10)	C(12)-C(13)-C(14)	120.3(3)
O(4)-P(2)-O(5)	110.15(10)	C(15)-C(14)-C(13)	120.1(3)
O(6)-P(2)-O(5)	97.58(9)	C(14)-C(15)-C(16)	120.3(2)
O(4)-P(2)-Pd(1)	113.90(8)	C(15)-C(16)-C(11)	119.0(2)
O(6)-P(2)-Pd(1)	107.68(7)	C(18)-C(17)-C(22)	121.1(3)
O(5)-P(2)-Pd(1)	113.97(7)	C(18)-C(17)-O(3)	122.6(3)
O(1A)-P(1A)-O(2A)	110.74(10)	C(22)-C(17)-O(3)	116.2(3)
O(1A)-P(1A)-O(3A)	105.59(10)	C(17)-C(18)-C(19)	118.6(4)
O(2A)-P(1A)-O(3A)	101.63(9)	C(20)-C(19)-C(18)	121.3(4)
O(1A)-P(1A)-Pd(2)	115.15(8)	C(19)-C(20)-C(21)	119.5(4)
O(2A)-P(1A)-Pd(2)	117.72(7)	C(20)-C(21)-C(22)	120.6(4)
O(3A)-P(1A)-Pd(2)	103.99(7)	C(17)-C(22)-C(21)	118.9(4)
O(4A)-P(2A)-O(6A)	112.07(10)	O(5)-C(23)-C(28)	117.3(2)
O(4A)-P(2A)-O(5A)	109.98(10)	O(5)-C(23)-C(24)	121.2(2)
O(6A)-P(2A)-O(5A)	98.55(9)	C(28)-C(23)-C(24)	121.2(2)
O(4A)-P(2A)-Pd(2)	116.65(8)	C(23)-C(24)-C(25)	118.5(3)
O(6A)-P(2A)-Pd(2)	106.48(6)	C(26)-C(25)-C(24)	120.5(3)
O(5A)-P(2A)-Pd(2)	111.60(7)	C(25)-C(26)-C(27)	120.4(3)
C(11)-O(2)-P(1)	123.57(14)	C(26)-C(27)-C(28)	120.0(3)
C(17)-O(3)-P(1)	122.40(15)	C(23)-C(28)-C(27)	119.3(3)
C(23)-O(5)-P(2)	128.66(16)	C(34)-C(29)-C(30)	121.4(2)
C(29)-O(6)-P(2)	121.74(15)	C(34)-C(29)-O(6)	117.0(2)
C(11A)-O(2A)-P(1A)	118.02(14)	C(30)-C(29)-O(6)	121.6(2)
C(17A)-O(3A)-P(1A)	127.42(15)	C(29)-C(30)-C(31)	119.2(3)
C(23A)-O(5A)-P(2A)	125.93(15)	C(30)-C(31)-C(32)	120.3(3)
C(29A)-O(6A)-P(2A)	126.10(15)	C(33)-C(32)-C(31)	119.8(3)
C(1)-N(1)-C(5)	117.8(2)	C(32)-C(33)-C(34)	120.3(3)
C(1)-N(1)-Pd(1)	126.92(17)	C(29)-C(34)-C(33)	119.0(2)
C(5)-N(1)-Pd(1)	115.14(16)	N(1A)-C(1A)-C(2A)	122.7(3)
C(10)-N(2)-C(6)	117.9(2)	C(3A)-C(2A)-C(1A)	119.0(3)
C(10)-N(2)-Pd(1)	126.88(18)	C(2A)-C(3A)-C(4A)	118.8(2)
C(6)-N(2)-Pd(1)	115.16(16)	C(3A)-C(4A)-C(5A)	120.0(3)
C(1A)-N(1A)-C(5A)	118.4(2)	N(1A)-C(5A)-C(4A)	120.9(3)
C(1A)-N(1A)-Pd(2)	126.98(17)	N(1A)-C(5A)-C(6A)	115.9(2)
C(5A)-N(1A)-Pd(2)	114.50(17)	C(4A)-C(5A)-C(6A)	123.1(2)
C(10A)-N(2A)-C(6A)	118.3(2)	N(2A)-C(6A)-C(7A)	121.4(3)
C(10A)-N(2A)-Pd(2)	126.92(18)	N(2A)-C(6A)-C(5A)	116.4(2)
C(6A)-N(2A)-Pd(2)	114.09(17)	C(7A)-C(6A)-C(5A)	122.2(2)
N(1)-C(1)-C(2)	123.3(2)	C(8A)-C(7A)-C(6A)	119.1(3)
C(3)-C(2)-C(1)	119.1(3)	C(9A)-C(8A)-C(7A)	119.5(3)
C(2)-C(3)-C(4)	118.5(3)	C(8A)-C(9A)-C(10A)	118.7(3)
C(5)-C(4)-C(3)	119.8(3)	N(2A)-C(10A)-C(9A)	122.9(3)

C(12A)-C(11A)-C(16A)	120.8(2)
C(12A)-C(11A)-O(2A)	117.8(2)
C(16A)-C(11A)-O(2A)	121.4(2)
C(11A)-C(12A)-C(13A)	119.1(2)
C(14A)-C(13A)-C(12A)	120.9(2)
C(13A)-C(14A)-C(15A)	119.5(2)
C(16A)-C(15A)-C(14A)	121.0(3)
C(15A)-C(16A)-C(11A)	118.8(2)
C(18A)-C(17A)-C(22A)	120.6(2)
C(18A)-C(17A)-O(3A)	123.4(2)
C(22A)-C(17A)-O(3A)	115.8(2)
C(17A)-C(18A)-C(19A)	119.0(3)
C(20A)-C(19A)-C(18A)	121.0(3)
C(19A)-C(20A)-C(21A)	119.5(3)
C(22A)-C(21A)-C(20A)	120.0(3)
C(21A)-C(22A)-C(17A)	119.8(3)
C(28A)-C(23A)-C(24A)	121.3(2)
C(28A)-C(23A)-O(5A)	121.6(2)
C(24A)-C(23A)-O(5A)	116.9(2)
C(25A)-C(24A)-C(23A)	119.2(2)
C(24A)-C(25A)-C(26A)	120.2(3)
C(27A)-C(26A)-C(25A)	119.8(3)
C(26A)-C(27A)-C(28A)	120.7(3)
C(23A)-C(28A)-C(27A)	118.7(3)
C(30A)-C(29A)-C(34A)	120.8(3)
C(30A)-C(29A)-O(6A)	117.9(2)
C(34A)-C(29A)-O(6A)	120.9(2)
C(29A)-C(30A)-C(31A)	118.7(3)
C(32A)-C(31A)-C(30A)	120.8(3)
C(33A)-C(32A)-C(31A)	119.6(3)
C(32A)-C(33A)-C(34A)	120.6(3)
C(29A)-C(34A)-C(33A)	119.4(3)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for compound **13**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Pd(1)	23(1)	20(1)	26(1)	1(1)	2(1)	0(1)
Pd(2)	25(1)	18(1)	26(1)	-1(1)	4(1)	1(1)
P(1)	21(1)	22(1)	25(1)	-1(1)	3(1)	1(1)
P(2)	27(1)	25(1)	23(1)	2(1)	3(1)	-1(1)
P(1A)	23(1)	21(1)	26(1)	-2(1)	3(1)	1(1)
P(2A)	26(1)	24(1)	24(1)	-2(1)	5(1)	1(1)
O(1)	23(1)	30(1)	37(1)	-2(1)	2(1)	-2(1)
O(2)	27(1)	22(1)	24(1)	-3(1)	0(1)	2(1)
O(3)	24(1)	32(1)	25(1)	-1(1)	4(1)	2(1)
O(4)	43(1)	33(1)	25(1)	5(1)	4(1)	4(1)
O(5)	29(1)	30(1)	31(1)	6(1)	11(1)	2(1)
O(6)	28(1)	27(1)	28(1)	-2(1)	5(1)	-4(1)
O(1A)	25(1)	27(1)	40(1)	-2(1)	5(1)	0(1)
O(2A)	29(1)	21(1)	26(1)	-3(1)	1(1)	3(1)
O(3A)	25(1)	30(1)	25(1)	-5(1)	4(1)	0(1)
O(4A)	44(1)	36(1)	25(1)	-1(1)	5(1)	8(1)
O(5A)	25(1)	25(1)	33(1)	2(1)	12(1)	1(1)
O(6A)	26(1)	27(1)	30(1)	-7(1)	9(1)	-2(1)
N(1)	26(1)	22(1)	32(1)	-1(1)	2(1)	0(1)
N(2)	26(1)	23(1)	31(1)	3(1)	2(1)	-2(1)
N(1A)	28(1)	20(1)	35(1)	-3(1)	8(1)	-2(1)
N(2A)	30(1)	21(1)	35(1)	4(1)	7(1)	-1(1)
C(1)	35(2)	25(1)	39(2)	-3(1)	-3(1)	3(1)
C(2)	47(2)	30(2)	34(2)	-5(1)	-2(1)	0(1)
C(3)	53(2)	34(2)	40(2)	-9(1)	5(1)	5(1)
C(4)	45(2)	32(2)	44(2)	-4(1)	4(1)	10(1)
C(5)	29(1)	22(1)	37(1)	0(1)	4(1)	0(1)
C(6)	31(1)	24(1)	36(1)	3(1)	3(1)	0(1)
C(7)	50(2)	27(1)	48(2)	-1(1)	-1(1)	11(1)
C(8)	49(2)	36(2)	54(2)	8(1)	-7(2)	13(1)
C(9)	41(2)	41(2)	39(2)	5(1)	-7(1)	3(1)
C(10)	37(2)	30(1)	34(1)	1(1)	-1(1)	0(1)
C(11)	22(1)	21(1)	27(1)	0(1)	3(1)	-1(1)
C(12)	36(1)	31(1)	24(1)	1(1)	7(1)	2(1)
C(13)	47(2)	36(2)	39(2)	-4(1)	13(1)	11(1)
C(14)	45(2)	35(2)	41(2)	4(1)	1(1)	15(1)
C(15)	39(2)	34(1)	26(1)	3(1)	-1(1)	3(1)
C(16)	31(1)	26(1)	24(1)	-2(1)	5(1)	-1(1)
C(17)	26(1)	40(2)	29(1)	15(1)	8(1)	6(1)
C(18)	39(2)	100(3)	46(2)	-22(2)	16(2)	-27(2)
C(19)	50(2)	177(5)	49(2)	-10(3)	9(2)	-57(3)
C(20)	27(2)	166(5)	74(3)	67(3)	11(2)	5(3)
C(21)	61(3)	74(3)	150(5)	68(3)	68(3)	42(2)
C(22)	61(2)	38(2)	111(3)	22(2)	56(2)	14(2)
C(23)	32(1)	16(1)	34(1)	1(1)	13(1)	1(1)
C(24)	45(2)	21(1)	31(1)	-2(1)	12(1)	1(1)
C(25)	64(2)	28(1)	42(2)	5(1)	30(2)	4(1)
C(26)	46(2)	39(2)	70(2)	9(2)	34(2)	-1(1)
C(27)	33(2)	42(2)	61(2)	9(2)	10(1)	-2(1)
C(28)	35(1)	29(1)	36(1)	3(1)	7(1)	0(1)
C(29)	25(1)	29(1)	28(1)	-2(1)	2(1)	-6(1)

C(30)	39(2)	37(2)	28(1)	3(1)	-2(1)	-7(1)
C(31)	38(2)	54(2)	26(1)	-9(1)	1(1)	-10(1)
C(32)	44(2)	37(2)	44(2)	-15(1)	13(1)	-10(1)
C(33)	51(2)	30(2)	42(2)	0(1)	14(1)	-6(1)
C(34)	41(2)	31(1)	28(1)	2(1)	6(1)	-4(1)
C(1A)	34(1)	25(1)	36(1)	-6(1)	6(1)	-4(1)
C(2A)	41(2)	28(1)	37(2)	-8(1)	9(1)	-8(1)
C(3A)	44(2)	26(1)	47(2)	-11(1)	19(1)	-7(1)
C(4A)	41(2)	23(1)	52(2)	-3(1)	16(1)	4(1)
C(5A)	33(1)	20(1)	40(2)	0(1)	12(1)	-1(1)
C(6A)	35(2)	21(1)	40(2)	3(1)	10(1)	1(1)
C(7A)	58(2)	28(2)	53(2)	3(1)	5(2)	13(1)
C(8A)	64(2)	37(2)	62(2)	13(2)	1(2)	21(2)
C(9A)	56(2)	38(2)	41(2)	13(1)	-2(1)	8(1)
C(10A)	41(2)	28(1)	37(2)	5(1)	6(1)	0(1)
C(11A)	25(1)	21(1)	28(1)	1(1)	3(1)	-1(1)
C(12A)	37(1)	29(1)	28(1)	-1(1)	10(1)	4(1)
C(13A)	33(1)	31(1)	41(2)	0(1)	12(1)	8(1)
C(14A)	33(1)	33(2)	41(2)	9(1)	4(1)	9(1)
C(15A)	39(2)	43(2)	31(1)	7(1)	9(1)	1(1)
C(16A)	28(1)	31(1)	35(1)	1(1)	10(1)	3(1)
C(17A)	25(1)	29(1)	26(1)	4(1)	5(1)	5(1)
C(18A)	33(1)	36(2)	30(1)	-4(1)	7(1)	-5(1)
C(19A)	31(2)	51(2)	40(2)	-3(1)	4(1)	-8(1)
C(20A)	25(1)	64(2)	45(2)	10(2)	8(1)	5(1)
C(21A)	31(2)	59(2)	44(2)	-3(2)	12(1)	16(1)
C(22A)	34(2)	41(2)	33(1)	-7(1)	5(1)	8(1)
C(23A)	30(1)	19(1)	33(1)	-2(1)	14(1)	-1(1)
C(24A)	33(1)	26(1)	30(1)	1(1)	10(1)	1(1)
C(25A)	30(1)	36(2)	43(2)	2(1)	8(1)	-3(1)
C(26A)	38(2)	43(2)	47(2)	0(1)	22(1)	-7(1)
C(27A)	52(2)	41(2)	32(1)	-3(1)	20(1)	-8(1)
C(28A)	37(1)	31(1)	28(1)	-7(1)	9(1)	-4(1)
C(29A)	28(1)	23(1)	32(1)	-3(1)	10(1)	-4(1)
C(30A)	28(2)	42(2)	99(3)	-31(2)	-1(2)	2(1)
C(31A)	34(2)	55(2)	156(4)	-53(3)	-2(2)	-10(2)
C(32A)	54(2)	31(2)	97(3)	-24(2)	23(2)	-12(2)
C(33A)	44(2)	29(2)	65(2)	-5(1)	16(2)	3(1)
C(34A)	32(1)	31(1)	47(2)	-3(1)	4(1)	-1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for compound **13**.

	x	y	z	U(eq)
H(1)	10463	8504	9189	42
H(2)	10164	8948	10043	47
H(3)	8442	9444	9761	51
H(4)	7093	9484	8603	49
H(7)	6096	9524	7543	52
H(8)	4933	9529	6360	59
H(9)	5357	9014	5641	51
H(10)	6928	8516	6111	41
H(12)	11571	6952	7391	36
H(13)	13215	6456	7826	48
H(14)	13926	6358	9036	50
H(15)	12991	6752	9813	40
H(16)	11325	7244	9385	32
H(18)	7878	7278	8259	73
H(19)	5380	7173	8172	111
H(20)	4070	7550	8793	107
H(21)	5208	8050	9495	106
H(22)	7664	8186	9542	77
H(24)	10614	7752	5432	38
H(25)	12739	7868	5004	50
H(26)	14964	7931	5776	59
H(27)	15107	7881	6981	54
H(28)	13001	7773	7420	40
H(30)	7513	7391	5437	43
H(31)	7551	6802	4837	48
H(32)	7937	6217	5459	50
H(33)	8431	6225	6684	48
H(34)	8426	6815	7289	40
H(1A)	11409	10451	4013	38
H(2A)	11276	10942	4817	42
H(3A)	9554	11437	4519	45
H(4A)	8028	11429	3406	45
H(7A)	6760	11404	2374	57
H(8A)	5503	11362	1204	67
H(9A)	6062	10860	494	56
H(10A)	7833	10410	965	43
H(12A)	12855	8921	2761	37
H(13A)	14560	8508	3436	41
H(14A)	14556	8407	4615	43
H(15A)	12853	8726	5133	45
H(16A)	11148	9144	4477	37
H(18A)	8172	9361	3141	40
H(19A)	5779	9246	3256	49
H(20A)	4839	9551	4135	53
H(21A)	6293	9982	4904	53
H(22A)	8671	10110	4779	43
H(24A)	13546	9627	2456	35
H(25A)	15707	9796	2106	43
H(26A)	15690	9927	924	49
H(27A)	13512	9899	99	48

H(28A)	11357	9710	435	38
H(30A)	6281	9076	1136	70
H(31A)	5895	8438	713	102
H(32A)	7789	7994	879	71
H(33A)	10077	8183	1450	55
H(34A)	10475	8810	1906	45

[1] Bruker-AXS. (2000-2003) SADABS V.2.05, SAINT V.6.22, SHELXTL V.6.10 & SMART 5.622 Software Reference Manuals. Bruker-AXS, Madison, Wisconsin, USA.

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