Journal of Organometallic Chemistry, 471 (1994) 259–263 259

Cyclometallated compounds of palladium(II) with diphosphines. The X-ray crystal structure of [{Pd[2,3,4-(MeO)$_3$C$_6$HC(H)=NCy]}$_2$(μ-Ph$_2$PC(=CH$_2$)PPh$_2$)Cl$_2$]

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(Received July 29, 1993)

Abstract

The reaction of the halide-bridged dimer complex [{Pd{2,3,4-(MeO)$_3$C$_6$HC(H)=NCy}Cl}$_2$] with Ph$_2$PC(=CH$_2$)PPh$_2$ (vdpp) in a dimer/diphosphine 1:1 molar ratio gives a yellow solid, from whose dichloromethane solution suitable crystals of [{Pd[2,3,4-(MeO)$_3$C$_6$HC(H)=NCy]}$_2${μ-Ph$_2$PC(=CH$_2$)PPh$_2$}Cl$_2$] (**1**) were grown. The structure of **1** has been determined by X-ray crystallography. In the structure there are two [{Pd[2,3,4-(MeO)$_3$C$_6$HC(H)=NCy]}Cl] units linked by a ditertiary diphosphine (vdpp) with each palladium atom showing a square-planar environment. Compound **1** is a non-electrolyte, as compared to a 1:1 electrolyte structure found for a similar complex.

Key words: Palladium; X-ray diffraction; Cyclometallation; Bridging ligand

1. Introduction

Cyclometallated compounds constitute an advanced area of organometallic chemistry [1–4]. The reactions of cyclometallated compounds proceed through the cleavage of one or more of the bonds to the metal atom, *e.g.* the metal–carbon bond, to yield organic products via insertion reactions [5], or of the metal-bridging ligand bonds, as in the reactions of halide-bridged dimer complexes with phosphines [6,7].

The reaction of cyclometallated Schiff base halide-bridged dimer complexes with ditertiary diphosphines in a dimer/diphosphine 1:1 molar ratio yields two types of dinuclear phosphine-bridged complex depending on the bite of the diphosphine: a) those complexes in which small bite diphosphines, such as Ph$_2$PCH$_2$PPh$_2$ (dppm) and Ph$_2$PC(=CH$_2$)PPh$_2$ (vdpp), bring the palladium atoms close together to allow them to be bridged also by a halogen atom, to give 1:1 dinuclear species [8]; b) those in which larger bite diphosphines, such as Ph$_2$PCH$_2$CH$_2$PPh$_2$ (dppe) and Ph$_2$P(CH$_2$)$_4$PPh$_2$ (dppb), prevent close approach of the metals, producing molecular species with terminal palladium–halogen bonds [9,10].

As a proof of this, we solved the crystal structure of one example of each kind, *i.e.*, one structure with a small bite diphosphine [{Pd[3-(MeO)C$_6$H$_3$C(H)=N Cy]}$_2$(μ-Ph$_2$PC(=CH$_2$)PPh$_2$)(μ-Cl)]Cl (**1**) [8] and one with a large bite diphosphine [{Pd[2,4-Me$_2$C$_6$H$_2$C(H)=NCy]}$_2$(μ-Ph$_2$P(CH$_2$)$_4$PPh$_2$)Cl$_2$] (**2**) [11]. Following our studies on such complexes we treated [{Pd{2,3,4-(MeO)$_3$C$_6$HC(H)=NCy}(Cl)]$_2$, with dppm and vdpp [11] and, on the basis of NMR data (in solution), we tentatively assigned the product the conformation (a) in Fig. 1; this was supported by conductivity data. Further, the $^2J(^{31}P-^{31}P)$ coupling constant for vdpp [12] suggests that the phosphine conformer expected in the complexes prepared previously [11] would be the one that appears in Fig. 1(a).

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0022-328X/94/$7.00
SSDI 0022-328X(93)24228-W

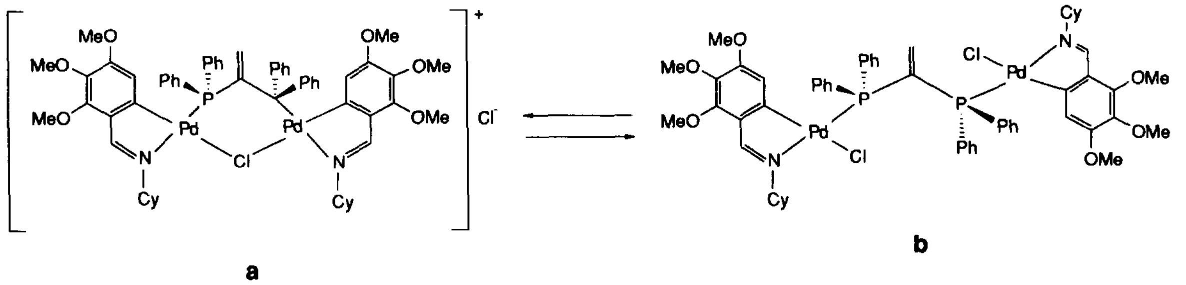

Fig. 1. Conformations of compound 1.

The crystal structure shows that the conformation in the solid state is actually (b) in Fig. 1. These results lead us to think that there may be an interconversion between these two conformations in solution when small bite diphosphines are used, as depicted in Fig. 1, and we are currently investigating the factors that favour (in the solid state) 1:1 electrolyte species of type (a) or molecular compounds of type (b). In the present paper we report the first crystal structure of a cyclometallated dinuclear palladium(II) complex with a small bite diphosphine in which the two metals centres are bonded only by diphosphine.

2. Results and discussion

Suitable crystals of the title compound were grown by slowly evaporating a dichloromethane solution. The molecular structure, with atom labelling, is illustrated in Fig. 2. Crystal data are given in Table 1. Fractional coordinates and isotropic thermal parameters are listed in Table 2, while bond lengths and bond angles with estimated standard deviations are listed in Tables 3 and 4, respectively.

The dinuclear molecule comprises two palladium(II) centres to each of which is bound a chelated *N*-(2,3,4-trimethoxybenzylidene)cyclohexylamine-C^6,N, a chlorine atom (*trans* to C^6) and a 1,1-bis(diphenylphosphino)ethene which bridges the two palladium atoms. The molecule exhibits approximate C_2 symmetry about the central $C{=}CH_2$ fragment. The crystal also contains a dichloromethane solvent molecule.

Each palladium adopts a tetrahedrally twisted, square-planar geometry (angles between the Pd, P, Cl and Pd, N, C planes are 12.2° and 9.9°, respectively) with the donor atoms of the chelating Schiff base occupying *cis* sites with a somewhat reduced bond angle [N(1)–Pd(1)–C(1) 79.1(8)° and N(2)–Pd(2)–C(31) 80.6(8)°] consequent upon chelation. This is reflected in the value of the Pd(1)–C(1)–C(2) and Pd(2)–C(31)–C(32) angles of 129.7(17)° and 131.7(16)°, respectively.

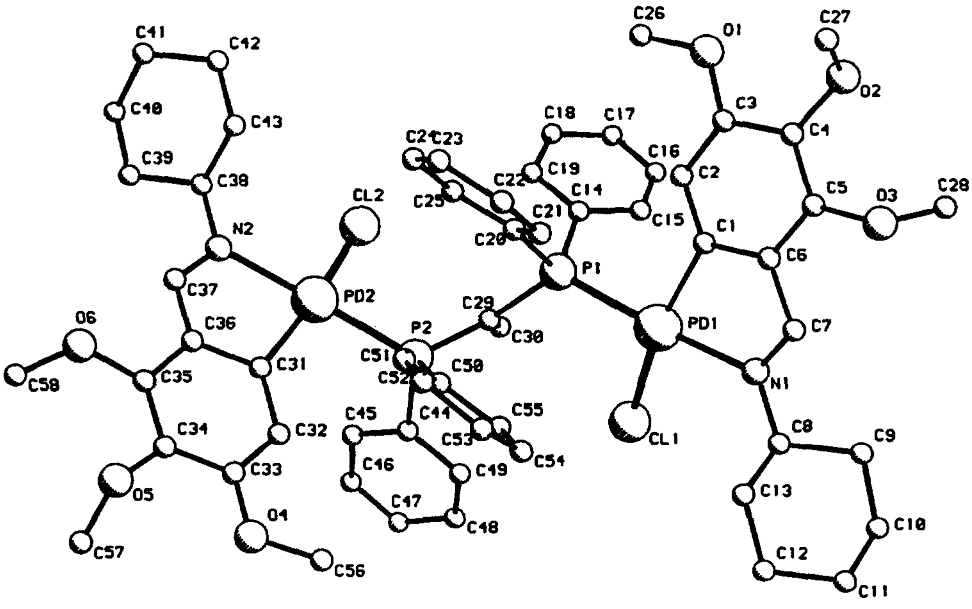

Fig. 2. The molecular structure of the title compound.

TABLE 1. Crystal data for $[\{Pd[2,3,4\text{-}(MeO)_3C_6HC(H)=NCy]Cl\}_2(\mu\text{-}Ph_2PC(=CH_2)PPh_2)]CH_2Cl_2$ (**1**)

Formula	$C_{59}H_{68}N_2O_6P_2Cl_4Pd_2$
Formula weight [a]	1315.6
Crystal colour	yellow
Crystal size (mm)	$0.9 \times 0.3 \times 0.2$
Crystal system	monoclinic
Space group	$P2_1/c$
Unit cell dimensions	
a (Å)	13.335(16)
b (Å)	24.433(20)
c (Å)	18.241(19)
β (°)	93.84(9)
Volume (Å^3)	5929(11)
Z	4
Density (calc.) g cm^{-1}	1.41
$F(000)$	2695.75
Radiation	Mo Kα ($\lambda = 0.71069$ Å)
Temperature (K)	297
μ(Mo Kα) (cm^{-1})	8.82
Scan type	2θ
Scan speed	3° min^{-1}
Scan range (°)	$3.5 < 2\theta < 50°$
Unique reflections	11293
No. of reflections ($I > 3.0\sigma(I)$)	2867
No. of parameters refined	381
R	0.0876
R_w	0.0759
GOF	1.472

[a] Includes solvent molecule.

The sum of angles about each palladium atom is 360.7°. The Pd–C bond lengths [Pd(1)–C(1) 2.014(22) Å and Pd(2)–C(31) 2.015(22) Å] are shorter than the expected value of 2.081 Å based on the sum of the covalent radii for carbon and palladium, 0.771 and 1.31 Å, respectively [13]. This shortening, observed elsewhere [7,10,14,15], is attributed to partial multiple-bond character of the Pd–C bond. The Pd–N bond lengths [Pd(1)–N(1) 2.123(19) Å and Pd(2)–N(2) 2.124(18) Å] are longer than the single bond value of 2.01 Å calculated from the covalent radii of N (sp^2) 0.70 Å; palladium 1.31 Å. This shows the *trans* influence of the phosphine. As in other cyclometallated complexes the C=N distances [N(1)–C(7) 1.264(29) Å and N(2)–C(37) 1.279(29) Å] are somewhat longer than the value of 1.237(3) Å observed for a related free donor [16], which is typical of a coordinated Schiff base [7,17].

The distances Pd(1)–P(1) 2.277(7) Å and Pd(2)–P(2) 2.269(7) Å are shorter than the sum of the single bond radii for palladium and phosphorus, 2.41 Å [13], suggesting that there may exist some double bonding between the palladium and phosphorus atoms and is similar to others found earlier [18]. The Pd–Cl bond

TABLE 2. Non-hydrogen atom coordinates ($\times 10^4$) and isotropic thermal parameters (Å$^2 \times 10^3$) for compound 1

Atom [a]	x	y	z	U_{eq} [b]
Pd(1)	3979(1)	1452(1)	4739(1)	32(1)
Pd(2)	963(1)	3650(1)	4822(1)	33(1)
P(1)	2504(5)	1880(2)	4935(4)	30(2)
P(2)	2466(5)	3214(3)	4950(4)	30(2)
Cl(1)	4945(5)	2037(3)	5562(4)	53(3)
Cl(2)	233(5)	3066(3)	5689(4)	49(3)
Cl(3)	1766(8)	−2442(5)	3592(6)	153(6)
Cl(4)	3066(11)	−2313(9)	2471(7)	266(12)
O(1)	1040(11)	21(6)	3680(9)	54(7)
O(2)	2407(14)	−668(8)	3173(11)	83(9)
O(3)	4389(12)	−283(6)	3111(9)	56(7)
O(4)	3549(11)	4899(6)	3288(10)	61(8)
O(5)	1970(13)	5449(7)	2594(9)	60(7)
O(6)	37(13)	5225(7)	3031(10)	68(8)
N(1)	5256(13)	1012(8)	4428(10)	42(8)
N(2)	−404(14)	4074(7)	4556(10)	40(8)
C(1)	3317(17)	815(9)	4198(12)	34(6)
C(2)	2330(16)	680(9)	4105(12)	32(6)
C(3)	2007(16)	207(9)	3743(12)	31(6)
C(4)	2698(18)	−145(10)	3457(13)	41(7)
C(5)	3695(18)	18(10)	3477(13)	41(7)
C(6)	3993(17)	487(9)	3842(13)	35(7)
C(7)	5023(16)	617(9)	4003(12)	28(6)
C(8)	6309(16)	1156(8)	4614(13)	35(7)
C(9)	7033(17)	666(9)	4636(13)	45(7)
C(10)	8085(20)	877(11)	4852(15)	68(9)
C(11)	8429(19)	1327(11)	4377(14)	64(8)
C(12)	7714(22)	1799(12)	4344(16)	86(10)
C(13)	6631(19)	1617(10)	4120(15)	66(9)
C(14)	1825(15)	1463(9)	5571(12)	32(6)
C(15)	2281(17)	1022(8)	5902(12)	33(6)
C(16)	1844(20)	715(11)	6428(15)	61(9)
C(17)	954(20)	846(11)	6655(15)	63(9)
C(18)	473(21)	1282(11)	6351(15)	74(9)
C(19)	913(19)	1607(11)	5777(15)	64(9)
C(20)	1728(16)	1993(8)	4069(12)	27(6)
C(21)	2040(17)	1848(9)	3413(12)	35(6)
C(22)	1471(19)	1967(10)	2781(15)	49(8)
C(23)	527(18)	2193(9)	2820(14)	42(7)
C(24)	186(18)	2354(9)	3478(13)	40(7)
C(25)	775(18)	2241(9)	4125(14)	41(7)
C(26)	302(20)	354(11)	3974(16)	80(10)
C(27)	1934(22)	−644(13)	2515(16)	106(12)
C(28)	4665(21)	−782(11)	3433(15)	81(10)
C(29)	2539(17)	2558(9)	5376(12)	30(6)
C(30)	2658(18)	2529(11)	6094(14)	58(8)
C(31)	1484(16)	4256(8)	4201(12)	29(6)
C(32)	2462(16)	4381(8)	4000(12)	32(6)
C(33)	2646(17)	4785(9)	3492(13)	34(6)
C(34)	1854(19)	5088(10)	3177(14)	47(8)
C(35)	885(20)	4985(11)	3358(15)	55(8)
C(36)	679(18)	4570(9)	3860(13)	38(7)
C(37)	−260(18)	4462(9)	4104(12)	41(7)
C(38)	−1414(16)	3941(8)	4786(13)	35(7)
C(39)	−2106(19)	4395(10)	4744(15)	62(9)
C(40)	−3131(19)	4249(10)	5002(15)	64(9)
C(41)	−3541(21)	3763(11)	4578(16)	78(10)

TABLE 2 (continued)

Atom [a]	x	y	z	U_{eq} [b]
C(42)	−2861(20)	3287(11)	4624(16)	77(10)
C(43)	−1813(18)	3438(9)	4396(14)	53(8)
C(44)	3302(17)	3613(10)	5597(12)	39(6)
C(45)	2898(24)	4046(12)	5916(17)	81(10)
C(46)	3483(27)	4315(15)	6498(19)	107(13)
C(47)	4414(25)	4165(13)	6657(17)	83(11)
C(48)	4818(27)	3715(14)	6375(19)	106(12)
C(49)	4279(22)	3450(12)	5778(16)	79(10)
C(50)	3043(17)	3120(9)	4122(13)	37(7)
C(51)	2544(19)	3222(9)	3444(14)	43(7)
C(52)	2989(18)	3161(10)	2787(14)	50(8)
C(53)	3951(19)	2974(9)	2785(15)	46(7)
C(54)	4482(21)	2842(10)	3439(15)	61(9)
C(55)	4028(17)	2906(9)	4089(14)	40(7)
C(56)	4333(22)	4565(12)	3527(17)	102(12)
C(57)	2416(22)	5940(11)	2802(16)	84(10)
C(58)	−98(22)	5767(12)	3096(17)	102(12)
C(59)	2785(25)	−2151(14)	3292(18)	114(13)

[a] Atoms C(59), Cl(3) and Cl(4) are from a dichloromethane molecule per asymmetric unit. [b] Equivalent isotropic U defined as one third of the trace of the orthogonalised U_{ij} tensor.

lengths [Pd(1)–Cl(1) 2.385(7) Å and Pd(2)–Cl(2) 2.386(7) Å] are consistent with Pd–Cl distances found in related species [10] but longer than the sum of the covalent radii (2.30 Å) as a consequence of the *trans* influence of the C(phenyl) atom. However, they are shorter than the values found for a similar structure with only one chlorine atom bridging two palladium atoms [8].

The aromatic rings are each planar (r.m.s. deviations 0.034 and 0.006 Å) with significant displacements of the methoxy substituents (maximum deviation from plane 0.237 Å). The cyclohexyl rings adopt chair conformations [r.m.s. deviations of central four-atom fragments 0.005 and 0.014; displacements of 1- and 4- sites +0.650, −0.626 and +0.587, −0.627 Å, respectively]. The four phenyl rings of the two diphenylphosphine units are each planar (r.m.s. deviations 0.006, 0.019, 0.033 and 0.015 Å).

The usual crystallographic data have been deposited with the Cambridge Crystallographic Data Centre.

3. Experimental details

The preparation of the title compound has been reported in an earlier paper [11].

3.1. *Single-crystal X-ray diffraction analysis*

Three-dimensional, room temperature X-ray data were collected in the range $3.5 < 2\theta < 50°$ on a Nicolet R3 diffractometer by the omega scan method. The 2867 independent reflections (of 11293 measured) for which $|F|/\sigma(|F|) > 5.0$ were corrected for Lorentz and polarisation effects, and corrected for absorption by eight azimuthal scans (minimum and maximum transmission coefficients 0.509 and 0.554). The structure was solved by direct methods and refined by blocked cascade least squares methods. Hydrogen atoms were included in calculated positions and refined in riding mode. Refinement converged at a final $R = 0.0876$ ($R_w = 0.0759$, 381 parameters, mean and maximum δ/σ 0.003, 0.013), with allowance for the

TABLE 3. Bond lengths (Å) with estimated standard deviations (e.s.d.s) for compound **1**

Pd(1)–P(1)	2.277(7)	Pd(1)–Cl(1)	2.385(7)
Pd(1)–N(1)	2.123(19)	Pd(1)–C(1)	2.014(22)
Pd(2)–P(2)	2.269(7)	Pd(2)–Cl(2)	2.386(7)
Pd(2)–N(2)	2.124(18)	Pd(2)–C(31)	2.015(22)
P(1)–C(14)	1.828(23)	P(1)–C(20)	1.852(22)
P(1)–C(29)	1.840(22)	P(2)–C(29)	1.781(22)
P(2)–C(44)	1.846(23)	P(2)–C(50)	1.754(25)
Cl(3)–C(59)	1.659(35)	Cl(4)–C(59)	1.617(37)
O(1)–C(3)	1.364(26)	O(1)–C(26)	1.409(31)
O(2)–C(4)	1.422(30)	O(2)–C(27)	1.319(35)
O(3)–C(5)	1.389(29)	O(3)–C(28)	1.392(30)
O(4)–C(33)	1.313(28)	O(4)–C(56)	1.374(33)
O(5)–C(34)	1.399(30)	O(5)–C(57)	1.381(31)
O(6)–C(35)	1.376(31)	O(6)–C(58)	1.342(33)
N(1)–C(7)	1.264(29)	N(1)–C(8)	1.466(27)
N(2)–C(37)	1.279(29)	N(2)–C(38)	1.474(28)
C(1)–C(2)	1.356(31)	C(1)–C(6)	1.398(33)
C(2)–C(3)	1.387(30)	C(3)–C(4)	1.388(33)
C(4)–C(5)	1.386(34)	C(5)–C(6)	1.371(33)
C(6)–C(7)	1.421(32)	C(8)–C(9)	1.536(30)
C(8)–C(13)	1.521(34)	C(9)–C(10)	1.521(34)
C(10)–C(11)	1.491(38)	C(11)–C(12)	1.496(39)
C(12)–C(13)	1.540(38)	C(14)–C(15)	1.360(30)
C(14)–C(19)	1.343(34)	C(15)–C(16)	1.377(35)
C(16)–C(17)	1.321(38)	C(17)–C(18)	1.344(37)
C(18)–C(19)	1.468(39)	C(20)–C(21)	1.340(32)
C(20)–C(25)	1.418(32)	C(21)–C(22)	1.367(34)
C(22)–C(23)	1.381(35)	C(23)–C(24)	1.370(36)
C(24)–C(25)	1.401(34)	C(29)–C(30)	1.311(35)
C(31)–C(32)	1.412(31)	C(31)–C(36)	1.429(31)
C(32)–C(33)	1.387(31)	C(33)–C(34)	1.383(33)
C(34)–C(35)	1.378(38)	C(35)–C(36)	1.404(36)
C(36)–C(37)	1.381(34)	C(38)–C(39)	1.442(33)
C(38)–C(43)	1.501(31)	C(39)–C(40)	1.517(37)
C(40)–C(41)	1.500(37)	C(41)–C(42)	1.473(38)
C(42)–C(43)	1.529(37)	C(44)–C(45)	1.338(39)
C(44)–C(49)	1.381(37)	C(45)–C(46)	1.434(46)
C(46)–C(47)	1.308(48)	C(47)–C(48)	1.342(48)
C(48)–C(49)	1.420(45)	C(50)–C(51)	1.389(34)
C(50)–C(55)	1.419(33)	C(51)–C(52)	1.380(37)
C(52)–C(53)	1.362(35)	C(53)–C(54)	1.383(37)
C(54)–C(55)	1.375(39)		

TABLE 4. Bond angles (°) with estimated standard deviations (e.s.d.s) for compound **1**

P(1)–Pd(1)–Cl(1)	93.6(2)	P(1)–Pd(1)–N(1)	172.4(5)
Cl(1)–Pd(1)–N(1)	93.6(5)	P(1)–Pd(1)–C(1)	94.4(7)
Cl(1)–Pd(1)–C(1)	166.2(6)	N(1)–Pd(1)–C(1)	79.1(8)
P(2)–Pd(2)–Cl(2)	92.7(2)	P(2)–Pd(2)–N(2)	172.5(6)
Cl(2)–Pd(2)–N(2)	93.5(5)	P(2)–Pd(2)–C(31)	93.9(6)
Cl(2)–Pd(2)–C(31)	169.5(7)	N(2)–Pd(2)–C(31)	80.6(8)
Pd(1)–P(1)–C(14)	108.3(7)	Pd(1)–P(1)–C(20)	111.9(7)
C(14)–P(1)–C(20)	110.7(10)	Pd(1)–P(1)–C(29)	119.0(8)
C(14)–P(1)–C(29)	102.9(10)	C(20)–P(1)–C(29)	103.6(10)
Pd(2)–P(2)–C(29)	119.3(8)	Pd(2)–P(2)–C(44)	108.0(8)
C(29)–P(2)–C(44)	100.5(11)	Pd(2)–P(2)–C(50)	114.1(8)
C(29)–P(2)–C(50)	104.1(11)	C(44)–P(2)–C(50)	109.8(11)
C(3)–O(1)–C(26)	117.1(18)	C(4)–O(2)–C(27)	113.2(21)
C(5)–O(3)–C(28)	115.3(19)	C(33)–O(4)–C(56)	118.5(20)
C(34)–O(5)–C(57)	114.0(19)	C(35)–O(6)–C(58)	119.6(21)
Pd(1)–N(1)–C(7)	112.5(15)	Pd(1)–N(1)–C(8)	126.1(14)
C(7)–N(1)–C(8)	121.2(19)	Pd(2)–N(2)–C(37)	110.2(15)
Pd(2)–N(2)–C(38)	127.8(14)	C(37)–N(2)–C(38)	121.8(19)
Pd(1)–C(1)–C(2)	129.7(17)	Pd(1)–C(1)–C(6)	113.3(16)
C(2)–C(1)–C(6)	116.9(20)	C(1)–C(2)–C(3)	122.0(21)
O(1)–C(3)–C(2)	125.4(20)	O(1)–C(3)–C(4)	114.2(19)
C(2)–C(3)–C(4)	120.2(20)	O(2)–C(4)–C(3)	121.4(21)
O(2)–C(4)–C(5)	120.4(21)	C(3)–C(4)–C(5)	118.1(21)
O(3)–C(5)–C(4)	120.2(21)	O(3)–C(5)–C(6)	119.8(21)
C(4)–C(5)–C(6)	120.0(22)	C(1)–C(6)–C(5)	122.0(22)
C(1)–C(6)–C(7)	114.9(20)	C(5)–C(6)–C(7)	122.1(21)
N(1)–C(7)–C(6)	119.4(21)	N(1)–C(8)–C(9)	114.1(17)
N(1)–C(8)–C(13)	110.1(18)	C(9)–C(8)–C(13)	112.9(19)
C(8)–C(9)–C(10)	108.1(19)	C(9)–C(10)–C(11)	114.4(21)
C(10)–C(11)–C(12)	111.8(23)	C(11)–C(12)–C(13)	111.8(23)
C(8)–C(14)–C(12)	110.7(21)	P(1)–C(14)–C(15)	119.9(17)
P(1)–C(13)–C(19)	121.9(18)	C(15)–C(14)–C(19)	118.0(22)
C(14)–C(15)–C(16)	122.9(22)	C(15)–C(16)–C(17)	121.2(25)
C(16)–C(17)–C(18)	118.5(26)	C(17)–C(18)–C(19)	121.3(26)
C(14)–C(19)–C(18)	118.1(23)	P(1)–C(20)–C(21)	122.3(17)
P(1)–C(20)–C(25)	117.0(17)	C(21)–C(20)–C(25)	120.7(21)
C(20)–C(21)–C(22)	120.6(22)	C(21)–C(22)–C(23)	119.9(24)
C(22)–C(23)–C(24)	121.2(23)	C(23)–C(24)–C(25)	118.7(22)
C(20)–C(25)–C(24)	118.6(22)	P(1)–C(29)–P(2)	128.3(13)
P(1)–C(29)–C(30)	112.7(18)	P(2)–C(29)–C(30)	119.0(18)
Pd(2)–C(31)–C(32)	131.7(16)	Pd(2)–C(31)–C(36)	111.2(16)
C(32)–C(31)–C(36)	116.8(20)	C(31)–C(32)–C(33)	122.5(20)
O(4)–C(33)–C(32)	123.2(20)	O(4)–C(33)–C(34)	117.3(21)
C(32)–C(33)–C(34)	119.5(22)	O(5)–C(34)–C(33)	122.2(22)
O(5)–C(34)–C(35)	116.8(22)	C(33)–C(34)–C(35)	120.3(23)
O(6)–C(35)–C(34)	125.0(23)	O(6)–C(35)–C(36)	113.6(22)
C(34)–C(35)–C(36)	121.1(23)	C(31)–C(36)–C(35)	119.8(22)
C(31)–C(36)–C(37)	115.3(21)	C(35)–C(36)–C(37)	124.7(22)
N(2)–C(37)–C(36)	121.9(21)	N(2)–C(38)–C(39)	114.1(19)
N(2)–C(38)–C(43)	110.3(18)	C(39)–C(38)–C(43)	113.6(19)
C(38)–C(39)–C(40)	112.9(21)	C(39)–C(40)–C(41)	109.3(22)
C(40)–C(41)–C(42)	113.2(23)	C(41)–C(42)–C(43)	111.4(22)
C(38)–C(43)–C(42)	111.7(21)	P(2)–C(44)–C(45)	116.9(19)
P(2)–C(44)–C(49)	121.4(20)	C(45)–C(44)–C(49)	121.6(24)
C(44)–C(45)–C(46)	118.0(28)	C(45)–C(46)–C(47)	119.8(32)
C(46)–C(47)–C(48)	123.0(32)	C(47)–C(48)–C(49)	118.1(30)
C(44)–C(49)–C(48)	118.3(27)	P(2)–C(50)–C(51)	122.1(19)
P(2)–C(50)–C(55)	123.1(18)	C(51)–C(50)–C(55)	114.7(22)
C(50)–C(51)–C(52)	123.1(23)	C(51)–C(52)–C(53)	119.9(23)
C(52)–C(53)–C(54)	120.2(25)	C(53)–C(54)–C(55)	119.2(25)
C(50)–C(55)–C(54)	122.7(23)	Cl(3)–C(59)–Cl(4)	116.4(20)

thermal anisotropy of Pd, P, Cl, O, and N only. Minimum and maximum final electron density -0.89 and 0.94 e Å^{-3}. A weighting scheme $w^{-1} = [\sigma^2(F) + 0.00050(F)^2]$ was used in the latter stages of refinement. Complex scattering factors were taken from ref. 19 and from the program package SHELXTL [20] as implemented on a Data General DG30 computer.

Acknowledgments

We wish to thank the Xunta de Galicia (Spain) (proyecto XUGA 20902A92) and SERC (UK) for support. J.J.F. and J.M.O. thank the Xunta de Galicia for grants.

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