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Supporting Information

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Polyfacetic Palladium Catalysts Towards the Tandem Diboration-Arylation Reaction of Alkenes

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General synthetic procedure for palladium complexes 2a-c. A sample of the corresponding palladium complex **1a-c** (0.042 mmol) was suspended in diethyl ether (6 mL) and then cooled to - 10 ºC. A slight excess of PhI·Cl_2 (13 mg, 0.055 mmol) in acetonitrile (1 mL) was added dropwise to the yellow suspension while the mixture was stirred. The colour of the suspension changed from yellow to red. The reaction mixture was stirred for 15 min at about - 5 ºC. The pale red-orange solution was decanted off and the red precipitate was washed with a 5:1 v/v mixture of diethyl ether/acetonitrile (3 × 3 mL) and then dried under vacuum to give the products in high yield (**2a**, 90%; **2b**, 96%; **2c**, 78%). Analytically pure samples were obtained by dissolving a sample in dichloromethane, and then filtering the solution and evaporating the solvent under vacuum. Finally the solid was washed with acetonitrile and dried under vacuum.

2a: $^{31}P\{^{1}H\}$ NMR ($CDCl_3$, 20 °C): -13.8 (s). ^{1}H NMR ($CDCl_3$, 20 °C): 8.22 (m, 2H), 7.74 (m, 1H), 7.42 (m, 4H), 7.20 (m, 4H), 6.99 (m, 1H), 6.80 (m, 1H), 6.74 (m, 1H), 1.14 (s, 3H). UV–Vis, $?_{max}$ nm (e, $M^{-1}\cdot mol^{-1}$): 499 (8.7×10^3), 434 (1.8×10^3), 379 (1.2×10^4). Anal. calcd. for $C_{40}H_{34}Cl_2P_2Pd_2O_4$: C 51.95, H 3.71. Found: C 51.51, H 3.63.

X-Ray Crystal Structure Data for compound 2a: $C_{40}H_{34}Cl_2O_4P_2Pd_2$, orthorhombic, space group *P-421/c*, *a*=22.299(3)Å, *b*=22.299(3)Å, *c*=17.250(3)Å, *a*=*b*=?=90°, *V*=8577(2)$Å^3$, *Z*=8; $Mo_{K\alpha}$ radiation, 273(2)K; 11321 reflections, 6135 independent; (μ = 1.073mm^{-1}); refinement (on F^2) with SHELXTL (version 6.1), 454 parameters, 0 restraints, R_1=0.0690 ($I>2\sigma$) and wR_2 (all data)=0.2020, GOF=1.129, max/min residual electron density: 2.152/-1.291 e $Å^{-3}$.

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loop_

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'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

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loop_

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Refinement of F^2^ against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2^, conventional R-factors R are based on F, with F set to zero for negative F^2^. The threshold expression of F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2^ are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

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Pd1 Pd 0.69731(6) 0.89626(6) 0.81290(6) 0.0365(4) Uani 1 1 d . . .

P1 P 0.6178(2) 0.8842(2) 0.7336(2) 0.0379(11) Uani 1 1 d . . .

Cl1 Cl 0.6499(2) 0.97072(19) 0.8986(2) 0.0483(11) Uani 1 1 d . . .

O1 O 0.7325(6) 0.9708(5) 0.7460(6) 0.051(3) Uani 1 1 d . . .

C1 C 0.7518(7) 0.9632(8) 0.6805(9) 0.043(4) Uani 1 1 d . . .

Pd2 Pd 0.75989(6) 0.83746(6) 0.71708(6) 0.0387(4) Uani 1 1 d . . .

P2 P 0.74997(19) 0.7558(2) 0.7945(2) 0.0425(11) Uani 1 1 d . . .

Cl2 Cl 0.8363(2) 0.7929(2) 0.6356(2) 0.0570(12) Uani 1 1 d . . .

O2 O 0.7614(5) 0.9147(5) 0.6492(5) 0.047(3) Uani 1 1 d . . .

C2 C 0.7644(11) 1.0226(9) 0.6375(10) 0.087(7) Uani 1 1 d . . .

H2A H 0.7777 1.0522 0.6740 0.131 Uiso 1 1 calc R . .

H2B H 0.7283 1.0362 0.6127 0.131 Uiso 1 1 calc R . .

H2C H 0.7949 1.0162 0.5992 0.131 Uiso 1 1 calc R . .

O3 O 0.7769(6) 0.9038(5) 0.8803(6) 0.055(3) Uani 1 1 d . . .

C3 C 0.8282(9) 0.9009(10) 0.8462(13) 0.066(6) Uani 1 1 d . . .

O4 O 0.8328(5) 0.8787(6) 0.7779(6) 0.057(3) Uani 1 1 d . . .

C4 C 0.8794(8) 0.9192(12) 0.8945(10) 0.091(8) Uani 1 1 d . . .

H4A H 0.9070 0.9422 0.8637 0.136 Uiso 1 1 calc R . .

H4B H 0.8993 0.8842 0.9142 0.136 Uiso 1 1 calc R . .

H4C H 0.8654 0.9433 0.9369 0.136 Uiso 1 1 calc R . .

C11 C 0.6350(8) 0.8257(8) 0.6666(8) 0.043(5) Uani 1 1 d . . .

C12 C 0.6884(8) 0.8056(8) 0.6555(7) 0.036(4) Uani 1 1 d . . .

C13 C 0.7004(8) 0.7636(8) 0.5992(8) 0.046(5) Uani 1 1 d . . .

H13 H 0.7391 0.7490 0.5930 0.055 Uiso 1 1 calc R . .

C14 C 0.6558(10) 0.7438(9) 0.5530(8) 0.062(6) Uani 1 1 d . . .

H14 H 0.6650 0.7176 0.5128 0.075 Uiso 1 1 calc R . .

C15 C 0.5952(9) 0.7617(9) 0.5641(9) 0.054(5) Uani 1 1 d . . .

H15 H 0.5640 0.7467 0.5340 0.065 Uiso 1 1 calc R . .

C16 C 0.5863(8) 0.8020(8) 0.6214(8) 0.048(5) Uani 1 1 d . . .

H16 H 0.5474 0.8147 0.6320 0.057 Uiso 1 1 calc R . .

C21 C 0.5463(6) 0.8653(8) 0.7829(8) 0.039(4) Uani 1 1 d . . .

C22 C 0.5296(7) 0.8080(8) 0.7965(8) 0.046(4) Uani 1 1 d . . .

H22 H 0.5544 0.7776 0.7782 0.055 Uiso 1 1 calc R . .

C23 C 0.4790(8) 0.7918(9) 0.8349(9) 0.053(5) Uani 1 1 d . . .

H23 H 0.4678 0.7518 0.8407 0.064 Uiso 1 1 calc R . .

C24 C 0.4458(9) 0.8371(14) 0.8643(11) 0.085(8) Uani 1 1 d . . .

H24 H 0.4114 0.8279 0.8925 0.102 Uiso 1 1 calc R . .

C25 C 0.4624(8) 0.9010(12) 0.8532(10) 0.066(6) Uani 1 1 d . . .

H25 H 0.4397 0.9321 0.8737 0.079 Uiso 1 1 calc R . .

C26 C 0.5101(7) 0.9107(7) 0.8133(8) 0.040(4) Uani 1 1 d . . .

H26 H 0.5212 0.9503 0.8042 0.048 Uiso 1 1 calc R . .

C31 C 0.6062(8) 0.9498(8) 0.6738(8) 0.043(4) Uani 1 1 d . . .

C32 C 0.6067(8) 1.0093(9) 0.7040(8) 0.054(5) Uani 1 1 d . . .

H32 H 0.6151 1.0145 0.7564 0.064 Uiso 1 1 calc R . .

C33 C 0.5959(10) 1.0568(10) 0.6620(10) 0.077(7) Uani 1 1 d . . .

H33 H 0.5985 1.0948 0.6842 0.092 Uiso 1 1 calc R . .

C34 C 0.5808(9) 1.0511(10) 0.5862(12) 0.073(6) Uani 1 1 d . . .

H34 H 0.5715 1.0846 0.5564 0.087 Uiso 1 1 calc R . .

C35 C 0.5796(10) 0.9923(11) 0.5532(10) 0.080(7) Uani 1 1 d . . .

H35 H 0.5705 0.9879 0.5009 0.096 Uiso 1 1 calc R . .

C36 C 0.5913(8) 0.9432(9) 0.5956(9) 0.051(5) Uani 1 1 d . . .

H36 H 0.5894 0.9053 0.5732 0.061 Uiso 1 1 calc R . .

C41 C 0.6888(8) 0.7645(7) 0.8586(8) 0.037(4) Uani 1 1 d . . .

C42 C 0.6671(7) 0.8239(7) 0.8725(7) 0.041(4) Uani 1 1 d . . .

C43 C 0.6247(7) 0.8326(7) 0.9329(7) 0.038(4) Uani 1 1 d . . .

H43 H 0.6121 0.8712 0.9447 0.046 Uiso 1 1 calc R . .

C44 C 0.6015(9) 0.7842(8) 0.9751(8) 0.050(5) Uani 1 1 d . . .

H44 H 0.5716 0.7907 1.0117 0.060 Uiso 1 1 calc R . .

C45 C 0.6229(7) 0.7268(9) 0.9626(9) 0.049(5) Uani 1 1 d . . .

H45 H 0.6102 0.6954 0.9939 0.059 Uiso 1 1 calc R . .

C46 C 0.6652(10) 0.7161(7) 0.9007(10) 0.062(6) Uani 1 1 d . . .

H46 H 0.6768 0.6771 0.8887 0.075 Uiso 1 1 calc R . .

C51 C 0.8169(8) 0.7436(8) 0.8534(8) 0.044(4) Uani 1 1 d . . .

C52 C 0.8720(9) 0.7528(10) 0.8264(10) 0.067(6) Uani 1 1 d . . .

H52 H 0.8778 0.7661 0.7758 0.081 Uiso 1 1 calc R . .

C53 C 0.9213(9) 0.7423(9) 0.8751(12) 0.068(6) Uani 1 1 d . . .

H53 H 0.9595 0.7509 0.8565 0.081 Uiso 1 1 calc R . .

C54 C 0.9161(10) 0.7211(10) 0.9448(12) 0.072(7) Uani 1 1 d . . .

H54 H 0.9499 0.7128 0.9745 0.087 Uiso 1 1 calc R . .

C55 C 0.8630(12) 0.7118(9) 0.9719(9) 0.073(7) Uani 1 1 d . . .

H55 H 0.8589 0.6972 1.0221 0.088 Uiso 1 1 calc R . .

C56 C 0.8112(9) 0.7231(8) 0.9281(10) 0.065(6) Uani 1 1 d . . .

H56 H 0.7734 0.7168 0.9495 0.078 Uiso 1 1 calc R . .

C61 C 0.7372(9) 0.6859(7) 0.7441(8) 0.044(5) Uani 1 1 d . . .

C62 C 0.7827(9) 0.6531(8) 0.7195(8) 0.057(5) Uani 1 1 d . . .

H62 H 0.8216 0.6653 0.7313 0.068 Uiso 1 1 calc R . .

C63 C 0.7745(12) 0.6022(10) 0.6774(12) 0.080(7) Uani 1 1 d . . .

H63 H 0.8076 0.5812 0.6589 0.095 Uiso 1 1 calc R . .

C64 C 0.7165(13) 0.5813(10) 0.6617(11) 0.082(7) Uani 1 1 d . . .

H64 H 0.7101 0.5455 0.6353 0.098 Uiso 1 1 calc R . .

C65 C 0.6705(10) 0.6154(10) 0.6865(10) 0.071(6) Uani 1 1 d . . .

H65 H 0.6316 0.6030 0.6756 0.085 Uiso 1 1 calc R . .

C66 C 0.6788(9) 0.6691(9) 0.7285(10) 0.061(5) Uani 1 1 d . . .

H66 H 0.6464 0.6921 0.7449 0.073 Uiso 1 1 calc R . .

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P1 0.037(3) 0.040(3) 0.036(2) 0.0007(18) -0.0011(18) 0.005(2)

Cl1 0.061(3) 0.040(3) 0.044(2) -0.0032(18) 0.007(2) 0.001(2)

O1 0.068(9) 0.039(8) 0.047(6) 0.002(5) 0.010(6) -0.001(6)

C1 0.044(11) 0.051(13) 0.034(9) -0.006(8) 0.017(8) -0.003(9)

Pd2 0.0366(8) 0.0430(9) 0.0365(6) 0.0020(6) -0.0003(6) 0.0021(6)

P2 0.040(3) 0.045(3) 0.042(2) 0.0028(19) -0.0022(19) 0.004(2)

Cl2 0.047(3) 0.071(3) 0.053(2) -0.002(2) 0.010(2) 0.017(3)

O2 0.055(8) 0.049(9) 0.037(5) 0.003(5) -0.002(5) -0.010(6)

C2 0.13(2) 0.083(18) 0.051(11) 0.010(11) 0.002(12) -0.017(16)

O3 0.059(9) 0.058(9) 0.048(6) 0.001(6) -0.013(6) 0.000(7)

C3 0.030(13) 0.073(16) 0.096(15) 0.027(12) 0.007(11) 0.002(11)

O4 0.031(7) 0.102(11) 0.038(6) -0.008(6) 0.004(5) -0.020(7)

C4 0.019(12) 0.18(3) 0.071(12) 0.000(14) -0.005(9) -0.011(13)

C11 0.047(13) 0.045(12) 0.039(9) -0.006(7) 0.001(8) 0.004(10)

C12 0.032(11) 0.043(11) 0.033(8) 0.002(7) -0.002(7) -0.005(9)

C13 0.040(11) 0.060(13) 0.038(8) -0.014(8) -0.009(8) -0.001(10)

C14 0.099(19) 0.050(13) 0.037(9) -0.014(8) 0.008(10) 0.011(14)

C15 0.059(15) 0.059(14) 0.045(9) -0.019(9) -0.018(9) 0.010(11)

C16 0.050(12) 0.048(12) 0.045(9) -0.003(9) 0.008(8) 0.004(10)

C21 0.017(9) 0.059(13) 0.040(8) 0.019(9) 0.006(7) -0.004(9)

C22 0.035(11) 0.056(13) 0.045(9) 0.001(9) 0.009(8) 0.000(9)

C23 0.045(13) 0.060(14) 0.055(10) 0.008(9) 0.008(9) 0.002(11)

C24 0.020(12) 0.17(3) 0.069(13) 0.025(16) 0.005(9) -0.007(15)

C25 0.010(10) 0.12(2) 0.067(11) -0.005(13) 0.003(9) 0.020(12)

C26 0.028(10) 0.042(11) 0.049(8) 0.007(8) 0.006(9) 0.002(8)

C31 0.036(11) 0.057(13) 0.034(9) 0.012(8) 0.001(7) 0.004(9)

C32 0.066(13) 0.065(14) 0.030(9) 0.013(9) -0.014(8) -0.002(11)

C33 0.12(2) 0.071(17) 0.042(11) 0.004(10) -0.005(11) -0.007(14)

C34 0.078(17) 0.049(15) 0.090(15) 0.014(12) 0.031(12) 0.001(12)

C35 0.092(18) 0.12(2) 0.034(9) 0.007(12) 0.007(10) 0.013(16)

C36 0.059(14) 0.054(13) 0.040(9) 0.012(9) 0.020(8) 0.007(10)

C41 0.053(12) 0.025(10) 0.032(7) 0.001(7) 0.000(7) -0.001(8)

C42 0.050(11) 0.040(11) 0.033(8) 0.009(7) -0.003(8) -0.010(9)

C43 0.047(11) 0.035(11) 0.033(8) -0.001(7) 0.001(7) -0.004(9)

C44 0.059(13) 0.056(14) 0.034(8) 0.012(8) 0.017(8) -0.006(11)

C45 0.033(12) 0.055(15) 0.061(10) 0.013(9) -0.006(8) -0.012(10)

C46 0.078(15) 0.021(10) 0.088(12) 0.014(9) 0.032(12) -0.004(10)

C51 0.040(12) 0.042(12) 0.050(9) 0.001(8) 0.009(8) 0.010(9)

C52 0.052(14) 0.095(17) 0.055(10) 0.002(11) -0.018(10) 0.019(12)

C53 0.040(13) 0.074(16) 0.089(15) -0.011(12) -0.003(11) 0.002(11)

C54 0.043(15) 0.10(2) 0.070(14) 0.011(12) -0.015(11) 0.020(13)

C55 0.11(2) 0.072(16) 0.038(10) 0.004(9) -0.024(12) 0.013(14)

C56 0.056(14) 0.074(16) 0.065(12) 0.005(10) -0.014(10) 0.007(11)

C61 0.062(14) 0.031(10) 0.040(8) 0.012(7) -0.001(8) 0.012(10)

C62 0.104(17) 0.029(11) 0.036(8) -0.002(8) 0.011(10) 0.027(11)

C63 0.085(19) 0.066(17) 0.088(14) 0.038(14) 0.019(13) 0.021(14)

C64 0.12(2) 0.063(16) 0.061(12) 0.002(10) -0.004(13) 0.023(17)

C65 0.062(15) 0.072(16) 0.079(12) -0.008(12) -0.010(11) -0.003(13)

C66 0.051(14) 0.057(14) 0.076(12) -0.008(10) 0.002(10) -0.013(10)

_geom_special_details

;

All esds (except the esd in the dihedral angle between two l.s. planes)

are estimated using the full covariance matrix. The cell esds are taken

into account individually in the estimation of esds in distances, angles

and torsion angles; correlations between esds in cell parameters are only

used when they are defined by crystal symmetry. An approximate (isotropic)

treatment of cell esds is used for estimating esds involving l.s. planes.

;

loop_

_geom_bond_atom_site_label_1

_geom_bond_atom_site_label_2

_geom_bond_distance

_geom_bond_site_symmetry_2

_geom_bond_publ_flag

Pd1 C42 2.028(15) . ?

Pd1 O3 2.127(11) . ?

Pd1 O1 2.170(11) . ?

Pd1 P1 2.255(4) . ?

Pd1 Cl1 2.462(4) . ?

Pd1 Pd2 2.5294(17) . ?

P1 C11 1.784(16) . ?

P1 C31 1.809(16) . ?

P1 C21 1.856(14) . ?

O1 C1 1.222(17) . ?

C1 O2 1.228(18) . ?

C1 C2 1.54(2) . ?

Pd2 C12 2.044(15) . ?

Pd2 O2 2.082(10) . ?

Pd2 O4 2.142(11) . ?

Pd2 P2 2.269(4) . ?

Pd2 Cl2 2.423(4) . ?

P2 C41 1.766(16) . ?

P2 C61 1.808(17) . ?

P2 C51 1.826(17) . ?

O3 C3 1.29(2) . ?

C3 O4 1.28(2) . ?

C3 C4 1.47(3) . ?

C11 C12 1.29(2) . ?

C11 C16 1.44(2) . ?

C12 C13 1.37(2) . ?

C13 C14 1.35(2) . ?

C14 C15 1.42(3) . ?

C15 C16 1.35(2) . ?

C21 C22 1.35(2) . ?

C21 C26 1.40(2) . ?

C22 C23 1.36(2) . ?

C23 C24 1.35(3) . ?

C24 C25 1.49(3) . ?

C25 C26 1.28(2) . ?

C31 C36 1.40(2) . ?

C31 C32 1.43(2) . ?

C32 C33 1.31(3) . ?

C33 C34 1.36(3) . ?

C34 C35 1.43(3) . ?

C35 C36 1.34(3) . ?

C41 C46 1.40(2) . ?

C41 C42 1.43(2) . ?

C42 C43 1.42(2) . ?

C43 C44 1.40(2) . ?

C44 C45 1.38(2) . ?

C45 C46 1.45(2) . ?

C51 C52 1.33(2) . ?

C51 C56 1.37(2) . ?

C52 C53 1.40(2) . ?

C53 C54 1.30(3) . ?

C54 C55 1.29(3) . ?

C55 C56 1.40(3) . ?

C61 C62 1.32(2) . ?

C61 C66 1.38(2) . ?

C62 C63 1.36(3) . ?

C63 C64 1.40(3) . ?

C64 C65 1.35(3) . ?

C65 C66 1.41(2) . ?

loop_

_geom_angle_atom_site_label_1

_geom_angle_atom_site_label_2

_geom_angle_atom_site_label_3

_geom_angle

_geom_angle_site_symmetry_1

_geom_angle_site_symmetry_3

_geom_angle_publ_flag

C42 Pd1 O3 93.6(5) . . ?

C42 Pd1 O1 177.2(6) . . ?

O3 Pd1 O1 85.9(5) . . ?

C42 Pd1 P1 87.2(4) . . ?

O3 Pd1 P1 175.0(3) . . ?

O1 Pd1 P1 93.0(3) . . ?

C42 Pd1 Cl1 95.1(5) . . ?

O3 Pd1 Cl1 88.7(3) . . ?

O1 Pd1 Cl1 87.6(3) . . ?

P1 Pd1 Cl1 96.18(16) . . ?

C42 Pd1 Pd2 95.9(5) . . ?

O3 Pd1 Pd2 86.4(3) . . ?

O1 Pd1 Pd2 81.4(3) . . ?

P1 Pd1 Pd2 88.57(11) . . ?

Cl1 Pd1 Pd2 168.22(12) . . ?

C11 P1 C31 104.6(7) . . ?

C11 P1 C21 108.4(8) . . ?

C31 P1 C21 108.8(8) . . ?

C11 P1 Pd1 108.2(6) . . ?

C31 P1 Pd1 111.2(6) . . ?

C21 P1 Pd1 115.1(5) . . ?

C1 O1 Pd1 120.9(11) . . ?

O1 C1 O2 126.1(16) . . ?

O1 C1 C2 113.0(16) . . ?

O2 C1 C2 120.9(14) . . ?

C12 Pd2 O2 90.5(5) . . ?

C12 Pd2 O4 174.9(6) . . ?

O2 Pd2 O4 84.7(5) . . ?

C12 Pd2 P2 87.2(4) . . ?

O2 Pd2 P2 174.9(4) . . ?

O4 Pd2 P2 97.5(3) . . ?

C12 Pd2 Cl2 96.0(5) . . ?

O2 Pd2 Cl2 90.1(3) . . ?

O4 Pd2 Cl2 85.8(3) . . ?

P2 Pd2 Cl2 94.61(16) . . ?

C12 Pd2 Pd1 95.1(5) . . ?

O2 Pd2 Pd1 87.0(3) . . ?

O4 Pd2 Pd1 82.9(3) . . ?

P2 Pd2 Pd1 88.73(12) . . ?

Cl2 Pd2 Pd1 168.49(13) . . ?

C41 P2 C61 106.0(8) . . ?

C41 P2 C51 107.4(7) . . ?

C61 P2 C51 105.5(8) . . ?

C41 P2 Pd2 110.8(5) . . ?

C61 P2 Pd2 115.1(5) . . ?

C51 P2 Pd2 111.6(6) . . ?

C1 O2 Pd2 118.7(10) . . ?

C3 O3 Pd1 119.2(12) . . ?

O4 C3 O3 120.6(18) . . ?

O4 C3 C4 124.4(18) . . ?

O3 C3 C4 114.7(19) . . ?

C3 O4 Pd2 123.7(12) . . ?

C12 C11 C16 119.3(15) . . ?

C12 C11 P1 123.4(14) . . ?

C16 C11 P1 117.2(13) . . ?

C11 C12 C13 121.6(15) . . ?

C11 C12 Pd2 121.5(13) . . ?

C13 C12 Pd2 116.9(12) . . ?

C14 C13 C12 119.8(16) . . ?

C13 C14 C15 121.9(15) . . ?

C16 C15 C14 115.1(16) . . ?

C15 C16 C11 122.0(17) . . ?

C22 C21 C26 117.4(14) . . ?

C22 C21 P1 122.1(13) . . ?

C26 C21 P1 120.3(13) . . ?

C21 C22 C23 124.4(17) . . ?

C24 C23 C22 116(2) . . ?

C23 C24 C25 122.2(18) . . ?

C26 C25 C24 116.0(18) . . ?

C25 C26 C21 123.9(18) . . ?

C36 C31 C32 116.8(15) . . ?

C36 C31 P1 120.0(14) . . ?

C32 C31 P1 123.0(11) . . ?

C33 C32 C31 123.5(14) . . ?

C32 C33 C34 120(2) . . ?

C33 C34 C35 118(2) . . ?

C36 C35 C34 121.9(18) . . ?

C35 C36 C31 119.1(19) . . ?

C46 C41 C42 119.9(14) . . ?

C46 C41 P2 121.9(13) . . ?

C42 C41 P2 117.9(12) . . ?

C43 C42 C41 118.3(14) . . ?

C43 C42 Pd1 119.0(12) . . ?

C41 C42 Pd1 122.6(11) . . ?

C44 C43 C42 121.5(16) . . ?

C45 C44 C43 120.3(16) . . ?

C44 C45 C46 119.6(15) . . ?

C41 C46 C45 119.9(16) . . ?

C52 C51 C56 117.8(17) . . ?

C52 C51 P2 122.4(12) . . ?

C56 C51 P2 119.8(14) . . ?

C51 C52 C53 119.2(17) . . ?

C54 C53 C52 123(2) . . ?

C55 C54 C53 118.5(19) . . ?

C54 C55 C56 122.1(18) . . ?

C51 C56 C55 119(2) . . ?

C62 C61 C66 120.9(18) . . ?

C62 C61 P2 120.7(16) . . ?

C66 C61 P2 118.3(14) . . ?

C61 C62 C63 122(2) . . ?

C62 C63 C64 120(2) . . ?

C65 C64 C63 117(2) . . ?

C64 C65 C66 123(2) . . ?

C61 C66 C65 116.9(19) . . ?

_diffrn_measured_fraction_theta_max 0.998

_diffrn_reflns_theta_full 23.25

_diffrn_measured_fraction_theta_full 0.998

_refine_diff_density_max 2.247

_refine_diff_density_min -1.310

_refine_diff_density_rms 0.392

2b: $^{31}P\{^{1}H\}$ NMR (CD_2Cl_2, 20 °C): -14.0 (s). ^{1}H NMR (CD_2Cl_2, 20 °C): 8.33 (m, 2H), 7.76 (m, 1H), 7.50 (m, 4H), 7.22 (m, 4H), 7.05 (m, 1H), 6.83 (m, 2H), 0.61 (s, 9H). UV–Vis, $?_{max}$ nm (e, $M^{-1}\cdot mol^{-1}$): 500 (1.4×10^4), 428 (3.9×10^3), 381 (2.0×10^4). Anal. calcd. for $C_{46}H_{46}Cl_2P_2Pd_2O_4$: C 53.99, H 4.12. Found: C 53.49, H 3.63.

X-Ray Crystal Structure Data for compound 2b: $C_{46}H_{46}Cl_2O_4P_2Pd_2$, orthorombic, space group *Pbna*, *a*=11.7110(3)Å, *b*=17.1950(4)Å, *c*=21.1460(7)Å, *a*=*b*=?=90°, *V*=4258.2(2)$Å^3$, *Z*=4; $Mo_{K\alpha}$ radiation, 293(2) K; 45790 reflections, 4528

independent; (μ = 1.088mm^{-1}); refinement (on F^2) with SHELXTL (version 6.1), 257parameters, 0 restraints, R_1=0.0487 (I>2σ) and wR_2 (all data)=0.1260, GOF= 1.031, max/min residual electron density: 0.605/-0.784 e Å^{-3}.

_audit_creation_method SHELXL-97

_chemical_formula_sum

'C46 H46 Cl2 O4 P2 Pd2'

_chemical_formula_weight 1008.47

loop_

_atom_type_symbol

_atom_type_description

_atom_type_scat_dispersion_real

_atom_type_scat_dispersion_imag

_atom_type_scat_source

'C' 'C' 0.0033 0.0016

'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

'H' 'H' 0.0000 0.0000

'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

'O' 'O' 0.0106 0.0060

'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

'Cl' 'Cl' 0.1484 0.1585

'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

'P' 'P' 0.1023 0.0942

'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

'Pd' 'Pd' -0.9988 1.0072

'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

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_symmetry_space_group_name_H-M Pbna

loop_

_symmetry_equiv_pos_as_xyz

'x, y, z'

'-x+1/2, -y, z+1/2'

'x, -y+1/2, -z'

'-x+1/2, y+1/2, -z+1/2'

'-x, -y, -z'

'x-1/2, y, -z-1/2'

'-x, y-1/2, z'

'x-1/2, -y-1/2, z-1/2'

_cell_length_a 11.7110(3)

_cell_length_b 17.1950(4)

_cell_length_c 21.1460(7)

_cell_angle_alpha 90.00

_cell_angle_beta 90.00

_cell_angle_gamma 90.00

_cell_volume 4258.2(2)

_cell_formula_units_Z 4

_cell_measurement_temperature 293(2)

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_cell_measurement_theta_min ?

_cell_measurement_theta_max ?

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_exptl_crystal_colour red-orange

_exptl_crystal_size_max 0.26

_exptl_crystal_size_mid 0.24

_exptl_crystal_size_min 0.20

_exptl_crystal_density_meas ?

_exptl_crystal_density_diffrn 1.573

_exptl_crystal_density_method 'not measured'

_exptl_crystal_F_000 2040

_exptl_absorpt_coefficient_mu 1.088

_exptl_absorpt_correction_type none

_exptl_absorpt_correction_T_min ?

_exptl_absorpt_correction_T_max ?

_exptl_absorpt_process_details ?

_exptl_special_details

;

?

;

_diffrn_ambient_temperature 293(2)

_diffrn_radiation_wavelength 0.71073

_diffrn_radiation_type MoK\a

_diffrn_radiation_source 'fine-focus sealed tube'

_diffrn_radiation_monochromator graphite

_diffrn_measurement_device_type ?

_diffrn_measurement_method ?

_diffrn_detector_area_resol_mean ?

_diffrn_standards_number ?

_diffrn_standards_interval_count ?

_diffrn_standards_interval_time ?

_diffrn_standards_decay_% ?

_diffrn_reflns_number 45790

_diffrn_reflns_av_R_equivalents 0.2523

_diffrn_reflns_av_sigmaI/netI 0.1202

_diffrn_reflns_limit_h_min -14

_diffrn_reflns_limit_h_max 14

_diffrn_reflns_limit_k_min -21

_diffrn_reflns_limit_k_max 21

_diffrn_reflns_limit_l_min -26

_diffrn_reflns_limit_l_max 26

_diffrn_reflns_theta_min 1.93

_diffrn_reflns_theta_max 26.73

_reflns_number_total 4528

_reflns_number_gt 2295

_reflns_threshold_expression >2sigma(I)

__computing_data_collection 'Collect(Nonius BV, 1997-2000)'

_computing_cell_refinement 'Denzo and Scalepack(Otwinowski & Minor1997)'

_computing_data_reduction 'Denzo and Scalepack(Otwinowski & Minor1997)'

_computing_structure_solution 'Bruker SHELXTL'

_computing_structure_refinement 'Bruker SHELXTL'

_computing_molecular_graphics 'Bruker SHELXTL'

_computing_publication_material 'Bruker SHELXTL'

_refine_special_details

;

Refinement of F^2^ against ALL reflections. The weighted R-factor wR and

goodness of fit S are based on F^2^, conventional R-factors R are based

on F, with F set to zero for negative F^2^. The threshold expression of

F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is

not relevant to the choice of reflections for refinement. R-factors based

on F^2^ are statistically about twice as large as those based on F, and R-

factors based on ALL data will be even larger.

;

_refine_ls_structure_factor_coef Fsqd

_refine_ls_matrix_type full

_refine_ls_weighting_scheme calc

_refine_ls_weighting_details

'calc w=1/[\s^2^(Fo^2^)+(0.0507P)^2^+0.6871P] where P=(Fo^2^+2Fc^2^)/3'

_atom_sites_solution_primary direct

_atom_sites_solution_secondary difmap

_atom_sites_solution_hydrogens geom

_refine_ls_hydrogen_treatment constr

_refine_ls_extinction_method SHELXL

_refine_ls_extinction_coef 0.00061(16)

_refine_ls_extinction_expression

'Fc^*^=kFc[1+0.001xFc^2^\l^3^/sin(2\q)]^-1/4^'

_refine_ls_number_reflns 4528

_refine_ls_number_parameters 257

_refine_ls_number_restraints 0

_refine_ls_R_factor_all 0.1418

_refine_ls_R_factor_gt 0.0487

_refine_ls_wR_factor_ref 0.1261

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_refine_ls_goodness_of_fit_ref 1.031

_refine_ls_restrained_S_all 1.031

_refine_ls_shift/su_max 0.001

_refine_ls_shift/su_mean 0.000

loop_

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_atom_site_type_symbol

_atom_site_fract_x

_atom_site_fract_y

_atom_site_fract_z

_atom_site_U_iso_or_equiv

_atom_site_adp_type

_atom_site_occupancy

_atom_site_symmetry_multiplicity

_atom_site_calc_flag

_atom_site_refinement_flags

_atom_site_disorder_assembly

_atom_site_disorder_group

Pd1 Pd 0.64520(4) 0.79982(2) 0.45617(2) 0.03425(17) Uani 1 1 d . . .

Cl1 Cl 0.68633(16) 0.88637(9) 0.36846(8) 0.0557(5) Uani 1 1 d . . .

P1 P 0.52327(15) 0.77795(9) 0.59762(8) 0.0391(4) Uani 1 1 d . . .

O1 O 0.7966(4) 0.7394(2) 0.4288(2) 0.0471(12) Uani 1 1 d . . .

C1 C 0.8168(6) 0.6719(4) 0.4481(3) 0.0462(18) Uani 1 1 d . . .

O2 O 0.7520(4) 0.6332(2) 0.4852(2) 0.0436(11) Uani 1 1 d . . .

C2 C 0.9271(7) 0.6319(4) 0.4267(4) 0.065(2) Uani 1 1 d . . .

C3 C 0.9020(8) 0.5534(4) 0.3996(5) 0.093(3) Uani 1 1 d . . .

H3A H 0.8565 0.5590 0.3621 0.139 Uiso 1 1 calc R . .

H3B H 0.8611 0.5229 0.4302 0.139 Uiso 1 1 calc R . .

H3C H 0.9724 0.5278 0.3892 0.139 Uiso 1 1 calc R . .

C4 C 0.9996(9) 0.6240(7) 0.4862(6) 0.131(4) Uani 1 1 d . . .

H4A H 1.0693 0.5973 0.4761 0.196 Uiso 1 1 calc R . .

H4B H 0.9582 0.5950 0.5174 0.196 Uiso 1 1 calc R . .

H4C H 1.0170 0.6747 0.5024 0.196 Uiso 1 1 calc R . .

C5 C 0.9883(9) 0.6802(5) 0.3771(6) 0.128(5) Uani 1 1 d . . .

H5A H 0.9444 0.6805 0.3388 0.192 Uiso 1 1 calc R . .

H5B H 1.0622 0.6580 0.3690 0.192 Uiso 1 1 calc R . .

H5C H 0.9972 0.7324 0.3922 0.192 Uiso 1 1 calc R . .

C11 C 0.4539(5) 0.8446(3) 0.5445(3) 0.0391(15) Uani 1 1 d . . .

C12 C 0.5048(5) 0.8589(3) 0.4856(3) 0.0339(15) Uani 1 1 d . . .

C13 C 0.4600(5) 0.9176(3) 0.4477(3) 0.0418(16) Uani 1 1 d . . .

H13 H 0.4918 0.9274 0.4082 0.050 Uiso 1 1 calc R . .

C14 C 0.3686(6) 0.9613(4) 0.4685(3) 0.0462(17) Uani 1 1 d . . .

H14 H 0.3420 1.0020 0.4436 0.055 Uiso 1 1 calc R . .

C15 C 0.3163(6) 0.9460(3) 0.5250(4) 0.0478(18) Uani 1 1 d . . .

H15 H 0.2535 0.9753 0.5374 0.057 Uiso 1 1 calc R . .

C16 C 0.3563(6) 0.8869(4) 0.5638(3) 0.0505(18) Uani 1 1 d . . .

H16 H 0.3199 0.8753 0.6018 0.061 Uiso 1 1 calc R . .

C21 C 0.5940(7) 0.8379(4) 0.6565(3) 0.0475(18) Uani 1 1 d . . .

C22 C 0.5564(7) 0.9137(3) 0.6693(3) 0.055(2) Uani 1 1 d . . .

H22 H 0.4997 0.9352 0.6440 0.066 Uiso 1 1 calc R . .

C23 C 0.5999(8) 0.9569(4) 0.7176(4) 0.075(3) Uani 1 1 d . . .

H23 H 0.5706 1.0061 0.7262 0.090 Uiso 1 1 calc R . .

C24 C 0.6878(8) 0.9269(4) 0.7533(4) 0.074(3) Uani 1 1 d . . .

H24 H 0.7202 0.9569 0.7852 0.089 Uiso 1 1 calc R . .

C25 C 0.7270(8) 0.8544(4) 0.7424(4) 0.075(3) Uani 1 1 d . . .

H25 H 0.7856 0.8343 0.7672 0.090 Uiso 1 1 calc R . .

C26 C 0.6800(7) 0.8097(4) 0.6941(3) 0.061(2) Uani 1 1 d . . .

H26 H 0.7074 0.7596 0.6872 0.073 Uiso 1 1 calc R . .

C31 C 0.4121(7) 0.7271(3) 0.6400(3) 0.0468(17) Uani 1 1 d . . .

C32 C 0.3087(6) 0.7095(4) 0.6119(4) 0.0554(19) Uani 1 1 d . . .

H32 H 0.2943 0.7259 0.5707 0.066 Uiso 1 1 calc R . .

C33 C 0.2261(7) 0.6677(4) 0.6444(4) 0.063(2) Uani 1 1 d . . .

H33 H 0.1574 0.6555 0.6247 0.076 Uiso 1 1 calc R . .

C34 C 0.2450(9) 0.6446(5) 0.7045(5) 0.078(3) Uani 1 1 d . . .

H34 H 0.1890 0.6169 0.7261 0.094 Uiso 1 1 calc R . .

C35 C 0.3459(9) 0.6615(4) 0.7338(4) 0.070(2) Uani 1 1 d . . .

H35 H 0.3574 0.6460 0.7754 0.083 Uiso 1 1 calc R . .

C36 C 0.4314(7) 0.7015(4) 0.7022(3) 0.0552(19) Uani 1 1 d . . .

H36 H 0.5009 0.7112 0.7220 0.066 Uiso 1 1 calc R . .

loop_

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_atom_site_aniso_U_22

_atom_site_aniso_U_33

_atom_site_aniso_U_23

_atom_site_aniso_U_13

_atom_site_aniso_U_12

Pd1 0.0361(3) 0.0325(2) 0.0342(3) -0.0002(2) 0.0012(3) -0.0008(2)

Cl1 0.0725(13) 0.0471(9) 0.0474(10) 0.0046(8) 0.0156(10) -0.0025(9)

P1 0.0466(11) 0.0387(9) 0.0321(9) -0.0013(7) -0.0006(9) 0.0030(8)

O1 0.047(3) 0.036(2) 0.059(3) 0.000(2) 0.015(2) 0.000(2)

C1 0.034(4) 0.046(4) 0.059(5) -0.012(4) 0.007(4) 0.000(3)

O2 0.041(3) 0.035(2) 0.055(3) 0.001(2) 0.007(2) -0.001(2)

C2 0.039(5) 0.053(4) 0.104(7) 0.001(4) 0.024(5) 0.014(4)

C3 0.086(7) 0.070(5) 0.123(8) -0.011(5) 0.055(6) 0.018(5)

C4 0.056(6) 0.147(10) 0.190(12) -0.018(9) -0.012(8) 0.042(7)

C5 0.092(8) 0.082(7) 0.210(13) 0.006(7) 0.090(9) 0.017(6)

C11 0.049(4) 0.035(3) 0.033(3) 0.001(3) 0.000(4) 0.001(3)

C12 0.037(4) 0.030(3) 0.034(3) -0.004(3) -0.005(3) -0.001(3)

C13 0.047(4) 0.039(3) 0.039(4) 0.006(3) -0.003(3) -0.005(3)

C14 0.041(4) 0.041(3) 0.057(5) 0.005(3) 0.000(4) 0.010(3)

C15 0.031(4) 0.039(3) 0.073(5) -0.010(4) -0.007(4) 0.010(3)

C16 0.051(4) 0.048(4) 0.052(4) -0.005(3) 0.012(4) 0.011(4)

C21 0.065(5) 0.043(4) 0.034(4) 0.004(3) -0.003(4) -0.005(4)

C22 0.074(6) 0.037(4) 0.054(5) -0.004(3) -0.016(4) 0.002(4)

C23 0.128(9) 0.039(4) 0.059(5) -0.012(4) -0.011(6) -0.003(5)

C24 0.114(8) 0.058(5) 0.052(5) -0.012(4) -0.020(5) -0.014(5)

C25 0.103(7) 0.068(5) 0.053(5) -0.014(4) -0.026(5) 0.003(5)

C26 0.090(6) 0.046(4) 0.047(4) -0.001(4) -0.017(4) -0.001(4)

C31 0.058(5) 0.041(3) 0.041(4) -0.004(3) 0.013(4) 0.004(3)

C32 0.054(5) 0.055(4) 0.057(5) -0.001(4) 0.019(4) -0.006(4)

C33 0.057(6) 0.063(5) 0.069(6) -0.011(4) 0.009(5) 0.003(4)

C34 0.082(7) 0.061(5) 0.092(8) 0.003(5) 0.034(6) -0.004(5)

C35 0.100(7) 0.062(5) 0.047(5) 0.008(4) 0.040(5) 0.010(5)

C36 0.071(5) 0.053(4) 0.042(4) 0.007(4) 0.016(4) 0.005(4)

_geom_special_details

;

All esds (except the esd in the dihedral angle between two l.s. planes)
are estimated using the full covariance matrix. The cell esds are taken
into account individually in the estimation of esds in distances, angles
and torsion angles; correlations between esds in cell parameters are only
used when they are defined by crystal symmetry. An approximate (isotropic)
treatment of cell esds is used for estimating esds involving l.s. planes.

;

loop_

_geom_bond_atom_site_label_1

_geom_bond_atom_site_label_2

_geom_bond_distance

_geom_bond_site_symmetry_2

_geom_bond_publ_flag

Pd1 C12 2.030(6) . ?

Pd1 O2 2.104(4) 3_566 ?

Pd1 O1 2.135(4) . ?

Pd1 P1 2.2628(18) 3_566 ?

Pd1 Cl1 2.4264(17) . ?

Pd1 Pd1 2.5241(9) 3_566 ?

P1 C11 1.799(6) . ?

P1 C31 1.806(7) . ?

P1 C21 1.817(7) . ?

P1 Pd1 2.2629(18) 3_566 ?

O1 C1 1.252(8) . ?

C1 O2 1.278(7) . ?

C1 C2 1.533(9) . ?

O2 Pd1 2.104(4) 3_566 ?

C2 C3 1.496(10) . ?

C2 C5 1.517(11) . ?

C2 C4 1.524(13) . ?

C3 H3A 0.9600 . ?

C3 H3B 0.9600 . ?

C3 H3C 0.9600 . ?

C4 H4A 0.9600 . ?

C4 H4B 0.9600 . ?

C4 H4C 0.9600 . ?

C5 H5A 0.9600 . ?

C5 H5B 0.9600 . ?

C5 H5C 0.9600 . ?

C11 C12 1.402(8) . ?

C11 C16 1.415(8) . ?

C12 C13 1.391(8) . ?

C13 C14 1.380(8) . ?

C13 H13 0.9300 . ?

C14 C15 1.368(9) . ?

C14 H14 0.9300 . ?

C15 C16 1.389(9) . ?

C15 H15 0.9300 . ?

C16 H16 0.9300 . ?

C21 C26 1.373(9) . ?

C21 C22 1.401(8) . ?

C22 C23 1.361(9) . ?

C22 H22 0.9300 . ?

C23 C24 1.378(11) . ?

C23 H23 0.9300 . ?

C24 C25 1.348(10) . ?

C24 H24 0.9300 . ?

C25 C26 1.392(9) . ?

C25 H25 0.9300 . ?

C26 H26 0.9300 . ?

C31 C32 1.383(10) . ?

C31 C36 1.406(9) . ?

C32 C33 1.387(10) . ?

C32 H32 0.9300 . ?

C33 C34 1.351(11) . ?

C33 H33 0.9300 . ?

C34 C35 1.365(12) . ?

C34 H34 0.9300 . ?

C35 C36 1.386(10) . ?

C35 H35 0.9300 . ?

C36 H36 0.9300 . ?

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_geom_angle_atom_site_label_2

_geom_angle_atom_site_label_3

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_geom_angle_site_symmetry_1

_geom_angle_site_symmetry_3

_geom_angle_publ_flag

C12 Pd1 O2 91.6(2) . 3_566 ?

C12 Pd1 O1 177.5(2) . . ?

O2 Pd1 O1 86.12(17) 3_566 . ?

C12 Pd1 P1 86.48(17) . 3_566 ?

O2 Pd1 P1 174.08(13) 3_566 3_566 ?

O1 Pd1 P1 95.74(13) . 3_566 ?

C12 Pd1 Cl1 95.05(18) . . ?

O2 Pd1 Cl1 89.80(12) 3_566 . ?

O1 Pd1 Cl1 85.79(13) . . ?

P1 Pd1 Cl1 95.94(6) 3_566 . ?

C12 Pd1 Pd1 96.59(17) . 3_566 ?

O2 Pd1 Pd1 86.51(11) 3_566 3_566 ?

O1 Pd1 Pd1 82.45(12) . 3_566 ?

P1 Pd1 Pd1 88.17(5) 3_566 3_566 ?

Cl1 Pd1 Pd1 167.88(5) . 3_566 ?

C11 P1 C31 107.0(3) . . ?

C11 P1 C21 105.8(3) . . ?

C31 P1 C21 105.3(3) . . ?

C11 P1 Pd1 110.3(2) . 3_566 ?

C31 P1 Pd1 114.7(2) . 3_566 ?

C21 P1 Pd1 113.1(3) . 3_566 ?

C1 O1 Pd1 121.3(4) . . ?

O1 C1 O2 124.8(6) . . ?

O1 C1 C2 118.6(6) . . ?

O2 C1 C2 116.6(6) . . ?

C1 O2 Pd1 115.4(4) . 3_566 ?

C3 C2 C5 108.8(8) . . ?

C3 C2 C4 110.2(8) . . ?

C5 C2 C4 110.8(9) . . ?

C3 C2 C1 110.7(7) . . ?

C5 C2 C1 110.9(6) . . ?

C4 C2 C1 105.4(7) . . ?

C2 C3 H3A 109.5 . . ?

C2 C3 H3B 109.5 . . ?

H3A C3 H3B 109.5 . . ?

C2 C3 H3C 109.5 . . ?

H3A C3 H3C 109.5 . . ?

H3B C3 H3C 109.5 . . ?

C2 C4 H4A 109.5 . . ?

C2 C4 H4B 109.5 . . ?

H4A C4 H4B 109.5 . . ?

C2 C4 H4C 109.5 . . ?

H4A C4 H4C 109.5 . . ?

H4B C4 H4C 109.5 . . ?

C2 C5 H5A 109.5 . . ?

C2 C5 H5B 109.5 . . ?

H5A C5 H5B 109.5 . . ?

C2 C5 H5C 109.5 . . ?

H5A C5 H5C 109.5 . . ?

H5B C5 H5C 109.5 . . ?

C12 C11 C16 120.7(6) . . ?

C12 C11 P1 118.3(5) . . ?

C16 C11 P1 120.7(5) . . ?

C13 C12 C11 118.5(6) . . ?

C13 C12 Pd1 119.5(5) . . ?

C11 C12 Pd1 121.9(4) . . ?

C14 C13 C12 120.4(6) . . ?

C14 C13 H13 119.8 . . ?

C12 C13 H13 119.8 . . ?

C15 C14 C13 121.3(6) . . ?

C15 C14 H14 119.4 . . ?

C13 C14 H14 119.4 . . ?

C14 C15 C16 120.5(6) . . ?

C14 C15 H15 119.8 . . ?

C16 C15 H15 119.8 . . ?

C15 C16 C11 118.5(6) . . ?

C15 C16 H16 120.7 . . ?

C11 C16 H16 120.7 . . ?

C26 C21 C22 116.6(6) . . ?

C26 C21 P1 122.1(5) . . ?

C22 C21 P1 121.2(6) . . ?

C23 C22 C21 122.4(7) . . ?

C23 C22 H22 118.8 . . ?

C21 C22 H22 118.8 . . ?

C22 C23 C24 119.1(7) . . ?

C22 C23 H23 120.5 . . ?

C24 C23 H23 120.5 . . ?

C25 C24 C23 120.5(7) . . ?

C25 C24 H24 119.8 . . ?

C23 C24 H24 119.8 . . ?

C24 C25 C26 120.2(8) . . ?

C24 C25 H25 119.9 . . ?

C26 C25 H25 119.9 . . ?

C21 C26 C25 121.3(7) . . ?

C21 C26 H26 119.4 . . ?

C25 C26 H26 119.4 . . ?

C32 C31 C36 118.3(7) . . ?

C32 C31 P1 121.6(5) . . ?

C36 C31 P1 120.0(6) . . ?

C31 C32 C33 120.7(7) . . ?

C31 C32 H32 119.6 . . ?

C33 C32 H32 119.6 . . ?

C34 C33 C32 120.3(9) . . ?

C34 C33 H33 119.8 . . ?

C32 C33 H33 119.8 . . ?

C33 C34 C35 120.3(9) . . ?

C33 C34 H34 119.8 . . ?

C35 C34 H34 119.8 . . ?

C34 C35 C36 120.9(8) . . ?

C34 C35 H35 119.6 . . ?

C36 C35 H35 119.6 . . ?

C35 C36 C31 119.3(8) . . ?

C35 C36 H36 120.3 . . ?

C31 C36 H36 120.3 . . ?

loop_

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_geom_torsion_atom_site_label_2

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_geom_torsion_site_symmetry_2

_geom_torsion_site_symmetry_3

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C12 Pd1 O1 C1 86(5) . . . . ?

O2 Pd1 O1 C1 106.2(5) 3_566 . . . ?

P1 Pd1 O1 C1 -68.2(5) 3_566 . . . ?

Cl1 Pd1 O1 C1 -163.7(5) . . . . ?

Pd1 Pd1 O1 C1 19.2(5) 3_566 . . . ?

Pd1 O1 C1 O2 -0.3(9) . . . . ?

Pd1 O1 C1 C2 -180.0(5) . . . . ?

O1 C1 O2 Pd1 -25.1(9) . . . 3_566 ?

C2 C1 O2 Pd1 154.6(5) . . . 3_566 ?

O1 C1 C2 C3 -128.0(7) . . . . ?

O2 C1 C2 C3 52.4(10) . . . . ?

O1 C1 C2 C5 -7.0(11) . . . . ?

O2 C1 C2 C5 173.3(8) . . . . ?

O1 C1 C2 C4 113.0(8) . . . . ?

O2 C1 C2 C4 -66.7(9) . . . . ?

C31 P1 C11 C12 -145.4(5) . . . . ?

C21 P1 C11 C12 102.7(5) . . . . ?

Pd1 P1 C11 C12 -20.0(5) 3_566 . . . ?

C31 P1 C11 C16 40.3(6) . . . . ?

C21 P1 C11 C16 -71.6(6) . . . . ?

Pd1 P1 C11 C16 165.7(5) 3_566 . . . ?

C16 C11 C12 C13 2.5(9) . . . . ?

P1 C11 C12 C13 -171.8(4) . . . . ?

C16 C11 C12 Pd1 179.8(5) . . . . ?

P1 C11 C12 Pd1 5.5(7) . . . . ?

O2 Pd1 C12 C13 100.1(5) 3_566 . . . ?

O1 Pd1 C12 C13 120(5) . . . . ?

P1 Pd1 C12 C13 -85.5(5) 3_566 . . . ?

Cl1 Pd1 C12 C13 10.2(5) . . . . ?

Pd1 Pd1 C12 C13 -173.2(4) 3_566 . . . ?

O2 Pd1 C12 C11 -77.2(5) 3_566 . . . ?

O1 Pd1 C12 C11 -57(5) . . . . ?

P1 Pd1 C12 C11 97.2(5) 3_566 . . . ?

Cl1 Pd1 C12 C11 -167.1(5) . . . . ?

Pd1 Pd1 C12 C11 9.5(5) 3_566 . . . ?

C11 C12 C13 C14 1.0(9) . . . . ?

Pd1 C12 C13 C14 -176.4(5) . . . . ?

C12 C13 C14 C15 -3.2(10) . . . . ?

C13 C14 C15 C16 1.8(10) . . . . ?

C14 C15 C16 C11 1.7(10) . . . . ?

C12 C11 C16 C15 -3.8(9) . . . . ?

P1 C11 C16 C15 170.3(5) . . . . ?

C11 P1 C21 C26 -161.8(6) . . . . ?

C31 P1 C21 C26 85.1(7) . . . . ?

Pd1 P1 C21 C26 -40.9(7) 3_566 . . . ?

C11 P1 C21 C22 23.8(7) . . . . ?

C31 P1 C21 C22 -89.4(6) . . . . ?

Pd1 P1 C21 C22 144.6(5) 3_566 . . . ?

C26 C21 C22 C23 -1.8(11) . . . . ?

P1 C21 C22 C23 173.0(6) . . . . ?

C21 C22 C23 C24 3.2(13) . . . . ?

C22 C23 C24 C25 -2.7(14) . . . . ?

C23 C24 C25 C26 1.0(14) . . . . ?

C22 C21 C26 C25 -0.1(11) . . . . ?

P1 C21 C26 C25 -174.8(6) . . . . ?

C24 C25 C26 C21 0.4(13) . . . . ?

C11 P1 C31 C32 33.6(6) . . . . ?

C21 P1 C31 C32 145.8(5) . . . . ?

Pd1 P1 C31 C32 -89.2(6) 3_566 . . . ?

C11 P1 C31 C36 -149.1(5) . . . . ?

C21 P1 C31 C36 -36.8(6) . . . . ?

Pd1 P1 C31 C36 88.2(5) 3_566 . . . ?

C36 C31 C32 C33 0.0(10) . . . . ?

P1 C31 C32 C33 177.4(5) . . . . ?

C31 C32 C33 C34 1.1(11) . . . . ?

C32 C33 C34 C35 -0.6(12) . . . . ?

C33 C34 C35 C36 -1.1(12) . . . . ?

C34 C35 C36 C31 2.2(11) . . . . ?

C32 C31 C36 C35 -1.6(10) . . . . ?

P1 C31 C36 C35 -179.1(5) . . . . ?

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_diffrn_reflns_theta_full 26.73

_diffrn_measured_fraction_theta_full 1.000

_refine_diff_density_max 0.605

_refine_diff_density_min -0.784

_refine_diff_density_rms 0.196

2c: $^{31}P\{^{1}H\}$ NMR ($CDCl_3$, 20 °C): -8.8 (q, $^{5}J_{F-P}$ 1.2 Hz). ^{19}F NMR ($CDCl_3$, 20 °C): -74.2 (d, $^{5}J_{P-F}$ 1.2 Hz). ^{1}H NMR ($CDCl_3$, 20 °C): 8.25 (m, 1H), 8.21 (m, 1H), 8.04 (m, 1H), 7.59 (m, 2H), 7.49 (m, 2H), 7.45-7.26 (m, 4H), 7.14 (m, 1H), 6.97 (m, 1H), 6.84 (m, 1H).

X-Ray Crystal Structure Data for compound 2c.2CH$_2$Cl$_2$: $C_{42}H_{32}Cl_6F_6O_4P_2Pd_2$, triclinic, space group *P-1*, *a*=11.4873(9)Å, *b*=12.2811(10)Å, *c*=17.4297(14)Å, *a*=96.8940(10)°, *b*=91.5210(10)°, ?=111.8540(10)°, *V*=2258.9(3)Å^3, *Z*=2, $Mo_{K\alpha}$ radiation, 173(2)K; 20358 reflections, 10494 independent; (μ = 1.288mm^{-1}); refinement (on F^2) with SHELXTL (version 6.1), 559 parameters, 0 restraints, R_1=0.0541 ($I>2\sigma$) and wR_2 (all data)=0.1033, GOF=1.061, max/min residual electron density: 1.542/-1.161 -e Å^{-3}.

_audit_creation_method SHELXL-97

_chemical_formula_sum

'C42 H32 Cl6 F6 O4 P2 Pd2'

_chemical_formula_weight 1202.12

loop_

_atom_type_symbol

_atom_type_description

_atom_type_scat_dispersion_real

_atom_type_scat_dispersion_imag

_atom_type_scat_source

'C' 'C' 0.0033 0.0016

'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

'H' 'H' 0.0000 0.0000

'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

'O' 'O' 0.0106 0.0060

'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

'F' 'F' 0.0171 0.0103

'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

'P' 'P' 0.1023 0.0942

'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

'Cl' 'Cl' 0.1484 0.1585

'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

'Pd' 'Pd' -0.9988 1.0072

'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_symmetry_cell_setting Triclinic

_symmetry_space_group_name_H-M P-1

loop_

_symmetry_equiv_pos_as_xyz

'x, y, z'

'-x, -y, -z'

_cell_length_a 11.4873(9)

_cell_length_b 12.2811(10)

_cell_length_c 17.4297(14)

_cell_angle_alpha 96.8940(10)

_cell_angle_beta 91.5210(10)

_cell_angle_gamma 111.8540(10)

_cell_volume 2258.9(3)

_cell_formula_units_Z 2

_cell_measurement_temperature 173(2)

_cell_measurement_reflns_used 9548

_cell_measurement_theta_min 2.29

_cell_measurement_theta_max 29.06

_exptl_crystal_description plate

_exptl_crystal_colour red

_exptl_crystal_size_max 0.18

_exptl_crystal_size_mid 0.18

_exptl_crystal_size_min 0.08

_exptl_crystal_density_meas 0

_exptl_crystal_density_diffrn 1.767

_exptl_crystal_density_method 'not measured'

_exptl_crystal_F_000 1188

_exptl_absorpt_coefficient_mu 1.288

_exptl_absorpt_correction_type none

_exptl_absorpt_correction_T_min ?

_exptl_absorpt_correction_T_max ?

_exptl_absorpt_process_details ?

_exptl_special_details

;

?

;

_diffrn_ambient_temperature 173(2)

_diffrn_radiation_wavelength 0.71073

_diffrn_radiation_type MoK\a

_diffrn_radiation_source 'fine-focus sealed tube'

_diffrn_radiation_monochromator graphite

_diffrn_measurement_device_type 'CCD area detector'

_diffrn_measurement_method 'phi and omega scans'

_diffrn_detector_area_resol_mean ?

_diffrn_standards_number ?

_diffrn_standards_interval_count ?

_diffrn_standards_interval_time ?

_diffrn_standards_decay_% ?

_diffrn_reflns_number 20358

_diffrn_reflns_av_R_equivalents 0.0338

_diffrn_reflns_av_sigmaI/netI 0.0627

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_diffrn_reflns_limit_h_max 15

_diffrn_reflns_limit_k_min -16

_diffrn_reflns_limit_k_max 16

_diffrn_reflns_limit_l_min -22

_diffrn_reflns_limit_l_max 23

_diffrn_reflns_theta_min 1.80

_diffrn_reflns_theta_max 29.06

_reflns_number_total 10494

_reflns_number_gt 8296

_reflns_threshold_expression >2sigma(I)

_computing_data_collection 'Bruker SMART'

_computing_cell_refinement 'Bruker SMART'

_computing_data_reduction 'Bruker SAINT'

_computing_structure_solution 'SHELXS-97 (Sheldrick, 1990)'

_computing_structure_refinement 'SHELXL-97 (Sheldrick, 1997)'

_computing_molecular_graphics 'Bruker SHELXTL'

_computing_publication_material 'Bruker SHELXTL'

_refine_special_details

;

Refinement of F^2^ against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2^, conventional R-factors R are based on F, with F set to zero for negative F^2^. The threshold expression of F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based

on F^2^ are statistically about twice as large as those based on F, and R-

factors based on ALL data will be even larger.

;

_refine_ls_structure_factor_coef Fsqd

_refine_ls_matrix_type full

_refine_ls_weighting_scheme calc

_refine_ls_weighting_details

'calc w=1/[\s^2^(Fo^2^)+(0.0307P)^2^+6.3144P] where P=(Fo^2^+2Fc^2^)/3'

_atom_sites_solution_primary direct

_atom_sites_solution_secondary difmap

_atom_sites_solution_hydrogens geom

_refine_ls_hydrogen_treatment constr

_refine_ls_extinction_method none

_refine_ls_extinction_coef ?

_refine_ls_number_reflns 10494

_refine_ls_number_parameters 559

_refine_ls_number_restraints 0

_refine_ls_R_factor_all 0.0771

_refine_ls_R_factor_gt 0.0540

_refine_ls_wR_factor_ref 0.1033

_refine_ls_wR_factor_gt 0.0963

_refine_ls_goodness_of_fit_ref 1.061

_refine_ls_restrained_S_all 1.061

_refine_ls_shift/su_max 0.001

_refine_ls_shift/su_mean 0.000

loop_

_atom_site_label

_atom_site_type_symbol

_atom_site_fract_x

_atom_site_fract_y

_atom_site_fract_z

_atom_site_U_iso_or_equiv

_atom_site_adp_type

_atom_site_occupancy

_atom_site_symmetry_multiplicity

_atom_site_calc_flag

_atom_site_refinement_flags

_atom_site_disorder_assembly

_atom_site_disorder_group

Pd2 Pd 0.87786(3) 0.37637(3) 0.693092(18) 0.01553(8) Uani 1 1 d . . .

P2 P 0.82573(10) 0.49953(10) 0.77919(6) 0.0174(2) Uani 1 1 d . . .

Cl2 Cl 0.69392(11) 0.32526(10) 0.60461(6) 0.0259(2) Uani 1 1 d . . .

O1 O 1.0408(3) 0.2269(3) 0.71809(17) 0.0228(7) Uani 1 1 d . . .

C1 C 0.9978(4) 0.2107(4) 0.6497(3) 0.0232(10) Uani 1 1 d . . .

F1 F 1.0444(4) 0.1314(3) 0.53025(19) 0.0654(12) Uani 1 1 d . . .

Pd1 Pd 1.04731(3) 0.38431(3) 0.792999(18) 0.01580(8) Uani 1 1 d . . .

P1 P 1.18905(10) 0.51875(10) 0.73095(6) 0.0187(2) Uani 1 1 d . . .

Cl1 Cl 1.20208(11) 0.35124(11) 0.87333(7) 0.0270(2) Uani 1 1 d . . .

O2 O 0.9447(3) 0.2671(3) 0.61680(17) 0.0235(7) Uani 1 1 d . . .

F2 F 1.0754(4) 0.0555(3) 0.62804(19) 0.0526(9) Uani 1 1 d . . .

C2 C 1.0031(5) 0.1026(5) 0.5973(3) 0.0331(12) Uani 1 1 d . . .

O4 O 0.7718(3) 0.2116(3) 0.73813(17) 0.0226(7) Uani 1 1 d . . .

C3 C 0.8070(4) 0.1952(4) 0.8022(2) 0.0214(9) Uani 1 1 d . . .

F3 F 0.8885(4) 0.0184(3) 0 .5849(2) 0.0633(11) Uani 1 1 d . . .

Cl3 Cl 0.5049(2) 0.12334(17) 0.43193(12) 0.0698(5) Uani 1 1 d . . .

O3 O 0.9017(3) 0.2581(3) 0.84685(17) 0.0239(7) Uani 1 1 d . . .

C4 C 0.7237(5) 0.0808(4) 0.8320(3) 0.0301(11) Uani 1 1 d . . .

F4 F 0.7860(5) 0.0309(5) 0.8642(5) 0.163(4) Uani 1 1 d . . .

Cl4 Cl 0.6021(3) -0.01901(19) 0.33075(17) 0.0994(8) Uani 1 1 d . . .

C5 C 0.4733(7) -0.0171(6) 0.3816(4) 0.065(2) Uani 1 1 d . . .

H5A H 0.3983 -0.0398 0.3448 0.077 Uiso 1 1 calc R . .

H5B H 0.4548 -0.0756 0.4185 0.077 Uiso 1 1 calc R . .

F5 F 0.6497(6) 0.0042(4) 0.7818(3) 0.139(3) Uani 1 1 d . . .

Cl5 Cl 0.17174(19) -0.01265(17) 0.81410(14) 0.0783(6) Uani 1 1 d . . .

C6 C 0.0930(6) 0.0451(6) 0.8828(4) 0.0619(19) Uani 1 1 d . . .

H6A H 0.0058 -0.0128 0.8831 0.074 Uiso 1 1 calc R . .

H6B H 0.0886 0.1185 0.8672 0.074 Uiso 1 1 calc R . .

Cl6 Cl 0.16709(19) 0.0773(2) 0.97708(13) 0.0801(6) Uani 1 1 d . . .

F6 F 0.6585(7) 0.0989(4) 0.8846(4) 0.157(3) Uani 1 1 d . . .

C11 C 1.1103(4) 0.5872(4) 0.6739(2) 0.0193(9) Uani 1 1 d . . .

C12 C 0.9836(4) 0.5243(4) 0.6493(2) 0.0196(9) Uani 1 1 d . . .

C13 C 0.9296(5) 0.5625(4) 0.5916(3) 0.0252(10) Uani 1 1 d . . .

H13 H 0.8436 0.5202 0.5741 0.030 Uiso 1 1 calc R . .

C14 C 1.0001(5) 0.6622(4) 0.5592(3) 0.0308(11) Uani 1 1 d . . .

H14 H 0.9622 0.6864 0.5188 0.037 Uiso 1 1 calc R . .

C15 C 1.1244(5) 0.7267(5) 0.5845(3) 0.0334(12) Uani 1 1 d . . .

H15 H 1.1711 0.7958 0.5627 0.040 Uiso 1 1 calc R . .

C16 C 1.1803(5) 0.6897(4) 0.6421(3) 0.0296(11) Uani 1 1 d . . .

H16 H 1.2658 0.7336 0.6600 0.036 Uiso 1 1 calc R . .

C21 C 1.2626(4) 0.4473(4) 0.6615(2) 0.0221(9) Uani 1 1 d . . .

C22 C 1.3066(5) 0.3615(5) 0.6826(3) 0.0289(11) Uani 1 1 d . . .

H22 H 1.2948 0.3379 0.7327 0.035 Uiso 1 1 calc R . .

C23 C 1.3670(5) 0.3116(5) 0.6299(3) 0.0362(12) Uani 1 1 d . . .

H23 H 1.3970 0.2539 0.6442 0.043 Uiso 1 1 calc R . .

C24 C 1.3843(5) 0.3451(5) 0.5569(3) 0.0407(14) Uani 1 1 d . . .

H24 H 1.4269 0.3111 0.5214 0.049 Uiso 1 1 calc R . .

C25 C 1.3398(5) 0.4277(5) 0.5355(3) 0.0390(13) Uani 1 1 d . . .

H25 H 1.3509 0.4499 0.4851 0.047 Uiso 1 1 calc R . .

C26 C 1.2787(5) 0.4786(5) 0.5874(3) 0.0286(11) Uani 1 1 d . . .

H26 H 1.2477 0.5351 0.5721 0.034 Uiso 1 1 calc R . .

C31 C 1.3143(4) 0.6335(4) 0.7935(3) 0.0225(9) Uani 1 1 d . . .

C32 C 1.4201(5) 0.6132(5) 0.8182(3) 0.0309(11) Uani 1 1 d . . .

H32 H 1.4284 0.5403 0.8006 0.037 Uiso 1 1 calc R . .

C33 C 1.5136(5) 0.7003(5) 0.8686(3) 0.0379(13) Uani 1 1 d . . .

H33 H 1.5863 0.6872 0.8849 0.046 Uiso 1 1 calc R . .

C34 C 1.5009(5) 0.8045(5) 0.8947(3) 0.0409(14) Uani 1 1 d . . .

H34 H 1.5643 0.8627 0.9301 0.049 Uiso 1 1 calc R . .

C35 C 1.3973(6) 0.8264(5) 0.8704(3) 0.0402(13) Uani 1 1 d . . .

H35 H 1.3901 0.8997 0.8882 0.048 Uiso 1 1 calc R . .

C36 C 1.3033(5) 0.7404(4) 0.8196(3) 0.0295(11) Uani 1 1 d . . .

H36 H 1.2317 0.7548 0.8029 0.035 Uiso 1 1 calc R . .

C41 C 0.9534(4) 0.5749(4) 0.8520(2) 0.0192(9) Uani 1 1 d . . .

C42 C 1.0430(4) 0.5254(4) 0.8638(2) 0.0179(9) Uani 1 1 d . . .

C43 C 1.1309(4) 0.5737(4) 0.9279(3) 0.0245(10) Uani 1 1 d . . .

H43 H 1.1923 0.5411 0.9367 0.029 Uiso 1 1 calc R . .

C44 C 1.1289(5) 0.6685(4) 0.9783(3) 0.0286(11) Uani 1 1 d . . .

H44 H 1.1883 0.6994 1.0221 0.034 Uiso 1 1 calc R . .

C45 C 1.0421(5) 0.7193(4) 0.9662(3) 0.0278(10) Uani 1 1 d . . .

H45 H 1.0423 0.7851 1.0010 0.033 Uiso 1 1 calc R . .

C46 C 0.9551(5) 0.6731(4) 0.9027(3) 0.0259(10) Uani 1 1 d . . .

H46 H 0.8960 0.7083 0.8934 0.031 Uiso 1 1 calc R . .

C51 C 0.7864(4) 0.6108(4) 0.7382(2) 0.0203(9) Uani 1 1 d . . .

C52 C 0.8789(5) 0.7223(4) 0.7349(3) 0.0269(10) Uani 1 1 d . . .

H52 H 0.9625 0.7410 0.7557 0.032 Uiso 1 1 calc R . .

C53 C 0.8482(5) 0.8056(5) 0.7013(3) 0.0367(12) Uani 1 1 d . . .

H53 H 0.9107 0.8819 0.6994 0.044 Uiso 1 1 calc R . .

C54 C 0.7265(5) 0.7782(5) 0.6702(3) 0.0369(13) Uani 1 1 d . . .

H54 H 0.7061 0.8358 0.6472 0.044 Uiso 1 1 calc R . .

C55 C 0.6358(5) 0.6686(5) 0.6726(3) 0.0325(12) Uani 1 1 d . . .

H55 H 0.5528 0.6504 0.6512 0.039 Uiso 1 1 calc R . .

C56 C 0.6645(4) 0.5836(4) 0.7062(3) 0.0259(10) Uani 1 1 d . . .

H56 H 0.6015 0.5074 0.7073 0.031 Uiso 1 1 calc R . .

C61 C 0.6914(4) 0.4199(4) 0.8309(3) 0.0213(9) Uani 1 1 d . . .

C62 C 0.6887(5) 0.4575(5) 0.9089(3) 0.0274(10) Uani 1 1 d . . .

H62 H 0.7592 0.5207 0.9358 0.033 Uiso 1 1 calc R . .

C63 C 0.5831(5) 0.4028(5) 0.9478(3) 0.0346(12) Uani 1 1 d . . .

H63 H 0.5819 0.4288 1.0013 0.042 Uiso 1 1 calc R . .

C64 C 0.4800(5) 0.3112(5) 0.9095(3) 0.0355(13) Uani 1 1 d . . .

H64 H 0.4073 0.2753 0.9362 0.043 Uiso 1 1 calc R . .

C65 C 0.4829(5) 0.2718(5) 0.8322(3) 0.0343(12) Uani 1 1 d . . .

H65 H 0.4127 0.2076 0.8059 0.041 Uiso 1 1 calc R . .

C66 C 0.5883(4) 0.3258(4) 0.7924(3) 0.0267(10) Uani 1 1 d . . .

H66 H 0.5899 0.2985 0.7392 0.032 Uiso 1 1 calc R . .

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_atom_site_aniso_label

_atom_site_aniso_U_11

_atom_site_aniso_U_22

_atom_site_aniso_U_33

_atom_site_aniso_U_23

_atom_site_aniso_U_13

_atom_site_aniso_U_12

Pd2 0.01439(16) 0.01737(17) 0.01452(16) 0.00225(12) 0.00084(12) 0.00567(13)

P2 0.0146(5) 0.0203(6) 0.0175(5) 0.0024(4) 0.0022(4) 0.0068(4)

Cl2 0.0211(6) 0.0291(6) 0.0239(5) 0.0026(4) -0.0059(4) 0.0064(5)

O1 0.0268(18) 0.0239(17) 0.0208(16) 0.0019(13) 0.0012(13) 0.0135(14)

C1 0.023(2) 0.019(2) 0.026(2) 0.0060(18) 0.0081(19) 0.0053(19)

F1 0.137(4) 0.052(2) 0.0333(18) 0.0140(16) 0.038(2) 0.061(2)

Pd1 0.01406(16) 0.01838(17) 0.01515(16) 0.00315(12) 0.00157(12) 0.00608(13)

P1 0.0141(5) 0.0233(6) 0.0191(5) 0.0052(4) 0.0032(4) 0.0068(5)

Cl1 0.0256(6) 0.0335(6) 0.0259(6) 0.0063(5) -0.0027(5) 0.0154(5)

O2 0.0318(18) 0.0224(17) 0.0187(15) 0.0020(13) 0.0029(13) 0.0131(14)

F2 0.081(3) 0.049(2) 0.046(2) -0.0016(16) -0.0001(18) 0.048(2)

C2 0.049(3) 0.032(3) 0.027(3) 0.007(2) 0.007(2) 0.024(3)

O4 0.0218(17) 0.0225(17) 0.0209(16) 0.0041(13) -0.0005(13) 0.0051(13)

C3 0.024(2) 0.022(2) 0.021(2) 0.0055(18) 0.0059(18) 0.0102(19)

F3 0.060(3) 0.033(2) 0.084(3) -0.0200(18) -0.008(2) 0.0116(18)

Cl3 0.0742(13) 0.0545(11) 0.0809(13) 0.0012(9) 0.0024(10) 0.0274(10)

O3 0.0224(17) 0.0278(18) 0.0176(15) 0.0031(13) 0.0002(13) 0.0050(14)

C4 0.034(3) 0.026(3) 0.025(2) 0.007(2) 0.001(2) 0.003(2)

F4 0.059(3) 0.116(4) 0.312(9) 0.165(6) -0.024(4) -0.007(3)

Cl4 0.1007(19) 0.0549(12) 0.135(2) -0.0114(13) 0.0236(16) 0.0266(12)

C5 0.071(5) 0.057(4) 0.046(4) 0.002(3) -0.010(4) 0.004(4)

F5 0.191(6) 0.065(3) 0.059(3) 0.031(2) -0.038(3) -0.071(3)

Cl5 0.0550(12) 0.0566(12) 0.1164(18) 0.0099(11) 0.0091(11) 0.0138(9)

C6 0.039(4) 0.050(4) 0.094(6) 0.016(4) -0.006(4) 0.012(3)

Cl6 0.0548(12) 0.0890(15) 0.0884(15) 0.0389(12) 0.0023(10) 0.0103(10)

F6 0.211(7) 0.046(3) 0.181(6) 0.020(3) 0.165(6) 0.000(3)

C11 0.016(2) 0.024(2) 0.020(2) 0.0076(18) 0.0029(17) 0.0089(18)

C12 0.017(2) 0.026(2) 0.017(2) 0.0044(17) 0.0061(17) 0.0088(18)

C13 0.026(3) 0.029(3) 0.022(2) 0.0070(19) -0.0012(19) 0.012(2)

C14 0.031(3) 0.036(3) 0.028(3) 0.015(2) 0.003(2) 0.013(2)

C15 0.030(3) 0.032(3) 0.039(3) 0.021(2) 0.007(2) 0.008(2)

C16 0.025(3) 0.030(3) 0.032(3) 0.012(2) 0.003(2) 0.006(2)

C21 0.015(2) 0.033(3) 0.021(2) 0.0042(19) 0.0044(17) 0.0114(19)

C22 0.023(3) 0.038(3) 0.026(2) 0.004(2) 0.002(2) 0.012(2)

C23 0.033(3) 0.046(3) 0.037(3) -0.001(2) 0.001(2) 0.025(3)

C24 0.035(3) 0.064(4) 0.033(3) -0.004(3) 0.008(2) 0.031(3)

C25 0.029(3) 0.068(4) 0.023(3) 0.004(3) 0.004(2) 0.023(3)

C26 0.024(3) 0.040(3) 0.022(2) 0.003(2) 0.0013(19) 0.012(2)

C31 0.014(2) 0.029(3) 0.021(2) 0.0046(19) 0.0034(17) 0.0039(19)

C32 0.022(2) 0.042(3) 0.027(2) 0.006(2) 0.001(2) 0.010(2)

C33 0.025(3) 0.049(4) 0.030(3) 0.012(2) -0.002(2) 0.002(2)

C34 0.031(3) 0.039(3) 0.033(3) 0.006(2) -0.005(2) -0.009(2)

C35 0.044(3) 0.022(3) 0.041(3) 0.000(2) -0.001(3) -0.001(2)

C36 0.024(3) 0.029(3) 0.032(3) 0.005(2) 0.003(2) 0.006(2)

C41 0.014(2) 0.020(2) 0.019(2) 0.0036(17) 0.0018(17) 0.0017(17)

C42 0.016(2) 0.021(2) 0.015(2) 0.0025(16) 0.0057(16) 0.0052(17)

C43 0.022(2) 0.029(3) 0.021(2) 0.0040(19) 0.0017(19) 0.008(2)

C44 0.029(3) 0.033(3) 0.018(2) -0.0017(19) -0.0028(19) 0.006(2)

C45 0.032(3) 0.026(3) 0.022(2) -0.0037(19) 0.000(2) 0.009(2)

C46 0.026(3) 0.026(3) 0.026(2) -0.0009(19) 0.003(2) 0.012(2)

C51 0.021(2) 0.026(2) 0.017(2) 0.0023(18) 0.0026(17) 0.0120(19)

C52 0.024(2) 0.025(2) 0.033(3) 0.005(2) 0.002(2) 0.010(2)

C53 0.042(3) 0.028(3) 0.043(3) 0.011(2) 0.001(3) 0.014(2)

C54 0.045(3) 0.039(3) 0.037(3) 0.010(2) 0.000(2) 0.026(3)

C55 0.028(3) 0.048(3) 0.030(3) 0.010(2) -0.001(2) 0.022(2)

C56 0.022(2) 0.032(3) 0.027(2) 0.006(2) 0.0038(19) 0.012(2)

C61 0.014(2) 0.026(2) 0.028(2) 0.0092(19) 0.0040(18) 0.0096(18)

C62 0.025(2) 0.038(3) 0.021(2) 0.007(2) 0.0020(19) 0.012(2)

C63 0.028(3) 0.057(4) 0.024(2) 0.012(2) 0.008(2) 0.019(3)

C64 0.020(3) 0.057(4) 0.038(3) 0.025(3) 0.014(2) 0.018(2)

C65 0.018(2) 0.042(3) 0.038(3) 0.013(2) 0.002(2) 0.004(2)

C66 0.020(2) 0.027(3) 0.029(2) 0.005(2) 0.0039(19) 0.003(2)

_geom_special_details

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All esds (except the esd in the dihedral angle between two l.s. planes)

are estimated using the full covariance matrix. The cell esds are taken

into account individually in the estimation of esds in distances, angles

and torsion angles; correlations between esds in cell parameters are only

used when they are defined by crystal symmetry. An approximate (isotropic)

treatment of cell esds is used for estimating esds involving l.s. planes.

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_geom_bond_atom_site_label_1

_geom_bond_atom_site_label_2

_geom_bond_distance

_geom_bond_site_symmetry_2

_geom_bond_publ_flag

Pd2 C12 2.024(4) . ?

Pd2 O2 2.133(3) . ?

Pd2 O4 2.189(3) . ?

Pd2 P2 2.2529(11) . ?

Pd2 Cl2 2.4175(11) . ?

Pd2 Pd1 2.5434(5) . ?

P2 C41 1.796(4) . ?

P2 C51 1.805(4) . ?

P2 C61 1.819(4) . ?

O1 C1 1.242(5) . ?

O1 Pd1 2.175(3) . ?

C1 O2 1.255(5) . ?

C1 C2 1.541(7) . ?

F1 C2 1.311(6) . ?

Pd1 C42 2.021(4) . ?

Pd1 O3 2.141(3) . ?

Pd1 P1 2.2511(12) . ?

Pd1 Cl1 2.4141(11) . ?

P1 C11 1.798(4) . ?

P1 C31 1.812(5) . ?

P1 C21 1.817(4) . ?

F2 C2 1.310(6) . ?

C2 F3 1.329(7) . ?

O4 C3 1.242(5) . ?

C3 O3 1.253(5) . ?

C3 C4 1.536(6) . ?

Cl3 C5 1.740(7) . ?

C4 F5 1.241(6) . ?

C4 F6 1.250(7) . ?

C4 F4 1.261(7) . ?

Cl4 C5 1.751(8) . ?

Cl5 C6 1.757(8) . ?

C6 Cl6 1.761(7) . ?

C11 C12 1.396(6) . ?

C11 C16 1.406(6) . ?

C12 C13 1.382(6) . ?

C13 C14 1.390(7) . ?

C14 C15 1.378(7) . ?

C15 C16 1.385(7) . ?

C21 C26 1.389(6) . ?

C21 C22 1.407(6) . ?

C22 C23 1.384(7) . ?

C23 C24 1.381(8) . ?

C24 C25 1.377(8) . ?

C25 C26 1.386(7) . ?

C31 C36 1.389(7) . ?

C31 C32 1.396(6) . ?

C32 C33 1.392(7) . ?

C33 C34 1.367(8) . ?

C34 C35 1.382(8) . ?

C35 C36 1.393(7) . ?

C41 C42 1.399(6) . ?

C41 C46 1.401(6) . ?

C42 C43 1.397(6) . ?

C43 C44 1.379(6) . ?

C44 C45 1.382(7) . ?

C45 C46 1.384(7) . ?

C51 C52 1.395(6) . ?

C51 C56 1.396(6) . ?

C52 C53 1.385(7) . ?

C53 C54 1.388(7) . ?

C54 C55 1.368(8) . ?

C55 C56 1.390(6) . ?

C61 C62 1.388(6) . ?

C61 C66 1.394(6) . ?

C62 C63 1.388(7) . ?

C63 C64 1.379(8) . ?

C64 C65 1.383(7) . ?

C65 C66 1.394(7) . ?

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_geom_angle_atom_site_label_1

_geom_angle_atom_site_label_2

_geom_angle_atom_site_label_3

_geom_angle

_geom_angle_site_symmetry_1

_geom_angle_site_symmetry_3

_geom_angle_publ_flag

C12 Pd2 O2 92.35(15) . . ?

C12 Pd2 O4 176.51(15) . . ?

O2 Pd2 O4 84.49(12) . . ?

C12 Pd2 P2 85.63(12) . . ?

O2 Pd2 P2 174.60(9) . . ?

O4 Pd2 P2 97.39(9) . . ?

C12 Pd2 Cl2 96.23(13) . . ?

O2 Pd2 Cl2 89.96(9) . . ?

O4 Pd2 Cl2 85.27(8) . . ?

P2 Pd2 Cl2 95.22(4) . . ?

C12 Pd2 Pd1 95.98(12) . . ?

O2 Pd2 Pd1 86.60(8) . . ?

O4 Pd2 Pd1 82.39(8) . . ?

P2 Pd2 Pd1 88.63(3) . . ?

Cl2 Pd2 Pd1 167.44(3) . . ?

C41 P2 C51 107.6(2) . . ?

C41 P2 C61 105.9(2) . . ?

C51 P2 C61 106.1(2) . . ?

C41 P2 Pd2 109.81(15) . . ?

C51 P2 Pd2 114.96(14) . . ?

C61 P2 Pd2 112.02(15) . . ?

C1 O1 Pd1 118.5(3) . . ?

O1 C1 O2 129.3(4) . . ?

O1 C1 C2 116.8(4) . . ?

O2 C1 C2 113.8(4) . . ?

C42 Pd1 O3 94.04(14) . . ?

C42 Pd1 O1 176.72(15) . . ?

O3 Pd1 O1 82.99(12) . . ?

C42 Pd1 P1 85.57(12) . . ?

O3 Pd1 P1 175.72(9) . . ?

O1 Pd1 P1 97.30(9) . . ?

C42 Pd1 Cl1 95.94(12) . . ?

O3 Pd1 Cl1 89.25(9) . . ?

O1 Pd1 Cl1 85.43(8) . . ?

P1 Pd1 Cl1 95.03(4) . . ?

C42 Pd1 Pd2 95.92(12) . . ?

O3 Pd1 Pd2 87.41(8) . . ?

O1 Pd1 Pd2 82.59(8) . . ?

P1 Pd1 Pd2 88.39(3) . . ?

Cl1 Pd1 Pd2 167.87(3) . . ?

C11 P1 C31 108.9(2) . . ?

C11 P1 C21 105.0(2) . . ?

C31 P1 C21 107.2(2) . . ?

C11 P1 Pd1 109.65(15) . . ?

C31 P1 Pd1 114.82(15) . . ?

C21 P1 Pd1 110.78(16) . . ?

C1 O2 Pd2 115.0(3) . . ?

F2 C2 F1 108.0(4) . . ?

F2 C2 F3 106.7(4) . . ?

F1 C2 F3 108.6(5) . . ?

F2 C2 C1 113.1(4) . . ?

F1 C2 C1 110.8(4) . . ?

F3 C2 C1 109.5(4) . . ?

C3 O4 Pd2 119.1(3) . . ?

O4 C3 O3 129.7(4) . . ?

O4 C3 C4 116.1(4) . . ?

O3 C3 C4 114.2(4) . . ?

C3 O3 Pd1 114.5(3) . . ?

F5 C4 F6 106.4(6) . . ?

F5 C4 F4 106.2(6) . . ?

F6 C4 F4 102.5(6) . . ?

F5 C4 C3 114.9(4) . . ?

F6 C4 C3 112.7(4) . . ?

F4 C4 C3 113.1(5) . . ?

Cl3 C5 Cl4 110.5(4) . . ?

Cl5 C6 Cl6 112.8(4) . . ?

C12 C11 C16 120.3(4) . . ?

C12 C11 P1 118.3(3) . . ?

C16 C11 P1 120.1(3) . . ?

C13 C12 C11 118.9(4) . . ?

C13 C12 Pd2 119.0(3) . . ?

C11 C12 Pd2 122.2(3) . . ?

C12 C13 C14 120.5(5) . . ?

C15 C14 C13 121.0(4) . . ?

C14 C15 C16 119.4(5) . . ?

C15 C16 C11 119.9(5) . . ?

C26 C21 C22 119.1(4) . . ?

C26 C21 P1 120.4(4) . . ?

C22 C21 P1 120.5(3) . . ?

C23 C22 C21 119.6(5) . . ?

C24 C23 C22 120.6(5) . . ?

C25 C24 C23 120.0(5) . . ?

C24 C25 C26 120.3(5) . . ?

C25 C26 C21 120.3(5) . . ?

C36 C31 C32 119.6(4) . . ?

C36 C31 P1 120.2(4) . . ?

C32 C31 P1 120.2(4) . . ?

C33 C32 C31 119.7(5) . . ?

C34 C33 C32 120.2(5) . . ?

C33 C34 C35 120.9(5) . . ?

C34 C35 C36 119.6(5) . . ?

C31 C36 C35 120.1(5) . . ?

C42 C41 C46 119.9(4) . . ?

C42 C41 P2 118.9(3) . . ?

C46 C41 P2 120.7(3) . . ?

C43 C42 C41 118.8(4) . . ?

C43 C42 Pd1 118.9(3) . . ?

C41 C42 Pd1 122.3(3) . . ?

C44 C43 C42 120.4(4) . . ?

C43 C44 C45 121.1(4) . . ?

C44 C45 C46 119.2(4) . . ?

C45 C46 C41 120.5(4) . . ?

C52 C51 C56 119.7(4) . . ?

C52 C51 P2 120.2(3) . . ?

C56 C51 P2 120.0(4) . . ?

C53 C52 C51 119.6(5) . . ?

C52 C53 C54 120.4(5) . . ?

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C62 C61 C66 119.4(4) . . ?

C62 C61 P2 119.5(4) . . ?

C66 C61 P2 121.0(3) . . ?

C61 C62 C63 120.1(5) . . ?

C64 C63 C62 120.6(5) . . ?

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C61 C66 C65 119.8(5) . . ?

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C12 Pd2 P2 C41 -76.7(2) . . . . ?

O2 Pd2 P2 C41 -8.5(9) . . . . ?

O4 Pd2 P2 C41 101.58(17) . . . . ?

Cl2 Pd2 P2 C41 -172.53(15) . . . . ?

Pd1 Pd2 P2 C41 19.44(15) . . . . ?

C12 Pd2 P2 C51 44.8(2) . . . . ?

O2 Pd2 P2 C51 113.0(9) . . . . ?

O4 Pd2 P2 C51 -136.97(19) . . . . ?

Cl2 Pd2 P2 C51 -51.08(17) . . . . ?

Pd1 Pd2 P2 C51 140.89(17) . . . . ?

C12 Pd2 P2 C61 166.0(2) . . . . ?

O2 Pd2 P2 C61 -125.8(9) . . . . ?

O4 Pd2 P2 C61 -15.74(17) . . . . ?

Cl2 Pd2 P2 C61 70.15(16) . . . . ?

Pd1 Pd2 P2 C61 -97.88(15) . . . . ?

Pd1 O1 C1 O2 -7.4(7) . . . . ?

Pd1 O1 C1 C2 176.0(3) . . . . ?

C1 O1 Pd1 C42 85(3) . . . . ?

C1 O1 Pd1 O3 109.9(3) . . . . ?

C1 O1 Pd1 P1 -65.8(3) . . . . ?

C1 O1 Pd1 Cl1 -160.3(3) . . . . ?

C1 O1 Pd1 Pd2 21.6(3) . . . . ?

C12 Pd2 Pd1 C42 71.42(17) . . . . ?

O2 Pd2 Pd1 C42 163.43(14) . . . . ?

O4 Pd2 Pd1 C42 -111.68(14) . . . . ?

P2 Pd2 Pd1 C42 -14.04(12) . . . . ?

Cl2 Pd2 Pd1 C42 -122.19(19) . . . . ?

C12 Pd2 Pd1 O3 165.21(14) . . . . ?

O2 Pd2 Pd1 O3 -102.78(12) . . . . ?

O4 Pd2 Pd1 O3 -17.89(12) . . . . ?

P2 Pd2 Pd1 O3 79.75(9) . . . . ?

Cl2 Pd2 Pd1 O3 -28.40(17) . . . . ?

C12 Pd2 Pd1 O1 -111.53(15) . . . . ?

O2 Pd2 Pd1 O1 -19.52(12) . . . . ?

O4 Pd2 Pd1 O1 65.37(12) . . . . ?

P2 Pd2 Pd1 O1 163.01(9) . . . . ?

Cl2 Pd2 Pd1 O1 54.86(17) . . . . ?

C12 Pd2 Pd1 P1 -13.96(12) . . . . ?

O2 Pd2 Pd1 P1 78.06(9) . . . . ?

O4 Pd2 Pd1 P1 162.94(9) . . . . ?

P2 Pd2 Pd1 P1 -99.41(4) . . . . ?

Cl2 Pd2 Pd1 P1 152.43(15) . . . . ?

C12 Pd2 Pd1 Cl1 -120.6(2) . . . . ?

O2 Pd2 Pd1 Cl1 -28.62(18) . . . . ?

O4 Pd2 Pd1 Cl1 56.26(18) . . . . ?

P2 Pd2 Pd1 Cl1 153.91(16) . . . . ?

Cl2 Pd2 Pd1 Cl1 45.8(2) . . . . ?

C42 Pd1 P1 C11 -74.9(2) . . . . ?

O3 Pd1 P1 C11 9.9(12) . . . . ?

O1 Pd1 P1 C11 103.48(18) . . . . ?

Cl1 Pd1 P1 C11 -170.50(16) . . . . ?

Pd2 Pd1 P1 C11 21.15(16) . . . . ?

C42 Pd1 P1 C31 48.0(2) . . . . ?

O3 Pd1 P1 C31 132.9(12) . . . . ?

O1 Pd1 P1 C31 -133.57(19) . . . . ?

Cl1 Pd1 P1 C31 -47.55(17) . . . . ?

Pd2 Pd1 P1 C31 144.11(17) . . . . ?

C42 Pd1 P1 C21 169.6(2) . . . . ?

O3 Pd1 P1 C21 -105.5(12) . . . . ?

O1 Pd1 P1 C21 -11.98(17) . . . . ?

Cl1 Pd1 P1 C21 74.04(16) . . . . ?

Pd2 Pd1 P1 C21 -94.30(15) . . . . ?

O1 C1 O2 Pd2 -17.0(6) . . . . ?

C2 C1 O2 Pd2 159.6(3) . . . . ?

C12 Pd2 O2 C1 121.6(3) . . . . ?

O4 Pd2 O2 C1 -56.9(3) . . . . ?

P2 Pd2 O2 C1 53.8(11) . . . . ?

Cl2 Pd2 O2 C1 -142.1(3) . . . . ?

Pd1 Pd2 O2 C1 25.8(3) . . . . ?

O1 C1 C2 F2 -12.9(7) . . . . ?

O2 C1 C2 F2 170.0(4) . . . . ?

O1 C1 C2 F1 -134.4(5) . . . . ?

O2 C1 C2 F1 48.6(6) . . . . ?

O1 C1 C2 F3 105.9(5) . . . . ?

O2 C1 C2 F3 -71.2(5) . . . . ?

C12 Pd2 O4 C3 81(2) . . . . ?

O2 Pd2 O4 C3 105.9(3) . . . . ?

P2 Pd2 O4 C3 -69.0(3) . . . . ?

Cl2 Pd2 O4 C3 -163.7(3) . . . . ?

Pd1 Pd2 O4 C3 18.6(3) . . . . ?

Pd2 O4 C3 O3 -3.8(7) . . . . ?

Pd2 O4 C3 C4 176.7(3) . . . . ?

O4 C3 O3 Pd1 -18.5(6) . . . . ?

C4 C3 O3 Pd1 161.0(3) . . . . ?

C42 Pd1 O3 C3 120.6(3) . . . . ?

O1 Pd1 O3 C3 -58.0(3) . . . . ?

P1 Pd1 O3 C3 36.1(14) . . . . ?

Cl1 Pd1 O3 C3 -143.5(3) . . . . ?

Pd2 Pd1 O3 C3 24.9(3) . . . . ?

O4 C3 C4 F5 17.3(8) . . . . ?

O3 C3 C4 F5 -162.3(6) . . . . ?

O4 C3 C4 F6 -104.8(7) . . . . ?

O3 C3 C4 F6 75.7(7) . . . . ?

O4 C3 C4 F4 139.5(6) . . . . ?

O3 C3 C4 F4 -40.1(7) . . . . ?

C31 P1 C11 C12 -151.5(3) . . . . ?

C21 P1 C11 C12 94.0(4) . . . . ?

Pd1 P1 C11 C12 -25.1(4) . . . . ?

C31 P1 C11 C16 41.1(4) . . . . ?

C21 P1 C11 C16 -73.5(4) . . . . ?

Pd1 P1 C11 C16 167.5(3) . . . . ?

C16 C11 C12 C13 2.0(7) . . . . ?

P1 C11 C12 C13 -165.4(3) . . . . ?

C16 C11 C12 Pd2 -179.8(3) . . . . ?

P1 C11 C12 Pd2 12.7(5) . . . . ?

O2 Pd2 C12 C13 95.4(4) . . . . ?

O4 Pd2 C12 C13 120(2) . . . . ?

P2 Pd2 C12 C13 -89.6(4) . . . . ?

Cl2 Pd2 C12 C13 5.2(4) . . . . ?

Pd1 Pd2 C12 C13 -177.8(3) . . . . ?

O2 Pd2 C12 C11 -82.7(4) . . . . ?

O4 Pd2 C12 C11 -58(3) . . . . ?

P2 Pd2 C12 C11 92.3(4) . . . . ?

Cl2 Pd2 C12 C11 -172.9(3) . . . . ?

Pd1 Pd2 C12 C11 4.1(4) . . . . ?

C11 C12 C13 C14 -0.5(7) . . . . ?

Pd2 C12 C13 C14 -178.6(4) . . . . ?

C12 C13 C14 C15 -1.3(8) . . . . ?

C13 C14 C15 C16 1.5(8) . . . . ?

C14 C15 C16 C11 0.1(8) . . . . ?

C12 C11 C16 C15 -1.9(7) . . . . ?

P1 C11 C16 C15 165.3(4) . . . . ?

C11 P1 C21 C26 17.0(4) . . . . ?

C31 P1 C21 C26 -98.8(4) . . . . ?

Pd1 P1 C21 C26 135.3(4) . . . . ?

C11 P1 C21 C22 -164.5(4) . . . . ?

C31 P1 C21 C22 79.8(4) . . . . ?

Pd1 P1 C21 C22 -46.1(4) . . . . ?

C26 C21 C22 C23 1.3(7) . . . . ?

P1 C21 C22 C23 -177.3(4) . . . . ?

C21 C22 C23 C24 -0.2(8) . . . . ?

C22 C23 C24 C25 -0.8(9) . . . . ?

C23 C24 C25 C26 0.7(9) . . . . ?

C24 C25 C26 C21 0.4(8) . . . . ?

C22 C21 C26 C25 -1.4(7) . . . . ?

P1 C21 C26 C25 177.2(4) . . . . ?

C11 P1 C31 C36 29.9(4) . . . . ?

C21 P1 C31 C36 143.0(4) . . . . ?

Pd1 P1 C31 C36 -93.5(4) . . . . ?

C11 P1 C31 C32 -152.6(4) . . . . ?

C21 P1 C31 C32 -39.5(4) . . . . ?

Pd1 P1 C31 C32 84.0(4) . . . . ?

C36 C31 C32 C33 -0.1(7) . . . . ?

P1 C31 C32 C33 -177.6(4) . . . . ?

C31 C32 C33 C34 0.9(8) . . . . ?

C32 C33 C34 C35 -1.5(8) . . . . ?

C33 C34 C35 C36 1.1(8) . . . . ?

C32 C31 C36 C35 -0.3(7) . . . . ?

P1 C31 C36 C35 177.2(4) . . . . ?

C34 C35 C36 C31 -0.2(8) . . . . ?

C51 P2 C41 C42 -146.9(3) . . . . ?

C61 P2 C41 C42 100.0(4) . . . . ?

Pd2 P2 C41 C42 -21.1(4) . . . . ?

C51 P2 C41 C46 41.1(4) . . . . ?

C61 P2 C41 C46 -72.0(4) . . . . ?

Pd2 P2 C41 C46 166.9(3) . . . . ?

C46 C41 C42 C43 1.7(6) . . . . ?

P2 C41 C42 C43 -170.3(3) . . . . ?

C46 C41 C42 Pd1 -179.7(3) . . . . ?

P2 C41 C42 Pd1 8.2(5) . . . . ?

O3 Pd1 C42 C43 97.5(3) . . . . ?

O1 Pd1 C42 C43 122(2) . . . . ?

P1 Pd1 C42 C43 -86.8(3) . . . . ?

Cl1 Pd1 C42 C43 7.8(3) . . . . ?

Pd2 Pd1 C42 C43 -174.7(3) . . . . ?

O3 Pd1 C42 C41 -81.1(4) . . . . ?

O1 Pd1 C42 C41 -56(3) . . . . ?

P1 Pd1 C42 C41 94.7(3) . . . . ?

Cl1 Pd1 C42 C41 -170.7(3) . . . . ?

Pd2 Pd1 C42 C41 6.8(4) . . . . ?

C41 C42 C43 C44 0.0(7) . . . . ?

Pd1 C42 C43 C44 -178.7(3) . . . . ?

C42 C43 C44 C45 -1.2(7) . . . . ?

C43 C44 C45 C46 0.7(7) . . . . ?

C44 C45 C46 C41 1.1(7) . . . . ?

C42 C41 C46 C45 -2.3(7) . . . . ?

P2 C41 C46 C45 169.7(4) . . . . ?

C41 P2 C51 C52 30.1(4) . . . . ?

C61 P2 C51 C52 143.1(4) . . . . ?

Pd2 P2 C51 C52 -92.5(4) . . . . ?

C41 P2 C51 C56 -153.1(4) . . . . ?

C61 P2 C51 C56 -40.1(4) . . . . ?

Pd2 P2 C51 C56 84.3(4) . . . . ?

C56 C51 C52 C53 1.3(7) . . . . ?

P2 C51 C52 C53 178.1(4) . . . . ?

C51 C52 C53 C54 -0.7(8) . . . . ?

C52 C53 C54 C55 0.0(8) . . . . ?

C53 C54 C55 C56 0.0(8) . . . . ?

C54 C55 C56 C51 0.6(7) . . . . ?

C52 C51 C56 C55 -1.2(7) . . . . ?

P2 C51 C56 C55 -178.0(4) . . . . ?

C41 P2 C61 C62 22.6(4) . . . . ?

C51 P2 C61 C62 -91.6(4) . . . . ?

Pd2 P2 C61 C62 142.2(3) . . . . ?

C41 P2 C61 C66 -160.9(4) . . . . ?

C51 P2 C61 C66 85.0(4) . . . . ?

Pd2 P2 C61 C66 -41.2(4) . . . . ?

C66 C61 C62 C63 -1.1(7) . . . . ?

P2 C61 C62 C63 175.5(4) . . . . ?

C61 C62 C63 C64 -0.2(8) . . . . ?

C62 C63 C64 C65 1.4(8) . . . . ?

C63 C64 C65 C66 -1.3(8) . . . . ?

C62 C61 C66 C65 1.1(7) . . . . ?

P2 C61 C66 C65 -175.4(4) . . . . ?

C64 C65 C66 C61 0.1(8) . . . . ?

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_diffrn_reflns_theta_full 29.06

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_refine_diff_density_min -1.161

_refine_diff_density_rms 0.120

Synthesis of 2d: A suspension of **1a** (50 mg, 0.042 mmol) in diethyl ether (6 mL) was cooled to - 10 ºC. A slight excess of bromine (5 µL, 0.097 mmol) in ether (1 mL) was added dropwise while the mixture was stirred. The colour changed from yellow to dark red. The reaction mixture was stirred for 15 min at about - 5 ºC. The pale red solution was decanted off and the red precipitate was washed with diethyl ether (3 × 3 mL) and then dried under vacuum to give the product in high yield (59 mg, 98 %). Single crystals suitable for X-ray diffraction were obtained by diffusion of diethyl ether into a dichloromethane solution of the compound at -15 ºC.

$^{31}P\{^{1}H\}$ NMR ($CDCl_3$, 20 °C): -15.0 (s). ^{1}H NMR ($CDCl_3$, 20 °C): 8.41-8.35 (m, 4H), 8.08-8.04 (m, 2H), 7.05-7.55 (m, 16H), 7.00-6.95 (m, 2H), 6.89-6.84 (m, 2H), 6.81-6.74 (m, 2H), 1.23 (s, 6H).

X-Ray Crystal Structure Data for compound 2d: $C_{40}H_{34}Br_2O_4P_2Pd_2$, orthorhombic, space group *P-421/c*, *a*=22.565(3)Å, *b*=22.565(3)Å, *c*=17.279(4)Å, *a*=*b*=?=90°, *V*=8798(3)Å^3, *Z*=8; $Mo_{K\alpha}$ radiation, 293(2)K; 16930 reflections, 9894 independent; (μ = 2.742mm^{-1}); refinement (on F^2) with SHELXTL (version 6.1), 453 parameters, 0 restraints, R_1=0.0608 ($I>2\sigma$) and wR_2 (all data)=0.1791, GOF=1.049, max/min residual electron density: 1.658/-1.531 e Å^{-3}.

_audit_creation_method SHELXL-97

'C40 H34 Br2 O4 P2 Pd2'

_chemical_formula_weight 1013.23

loop_

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_atom_type_description

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'C' 'C' 0.0033 0.0016

'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

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'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

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'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

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loop_

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'x+1/2, -y+1/2, -z+1/2'

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'y+1/2, x+1/2, z+1/2'

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_cell_length_b 22.565(3)

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_exptl_crystal_density_method 'not measured'

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_computing_cell_refinement 'Denzo and Scalepack(Otwinowski & Minor1997)'

_computing_data_reduction 'Denzo and Scalepack(Otwinowski & Minor1997)'

_computing_structure_solution 'Bruker SHELXTL'

_computing_structure_refinement 'Bruker SHELXTL'

_computing_molecular_graphics 'Bruker SHELXTL'

_computing_publication_material 'Bruker SHELXTL'

_refine_special_details

;

Refinement of F^2^ against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2^, conventional R-factors R are based on F, with F set to zero for negative F^2^. The threshold expression of F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2^ are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

;

_refine_ls_structure_factor_coef Fsqd

_refine_ls_matrix_type full

_refine_ls_weighting_scheme calc

_refine_ls_weighting_details

'calc w=1/[\s^2^(Fo^2^)+(0.1136P)^2^+0.0000P] where P=(Fo^2^+2Fc^2^)/3'

_atom_sites_solution_primary direct

_atom_sites_solution_secondary difmap

_atom_sites_solution_hydrogens geom

_refine_ls_hydrogen_treatment constr

_refine_ls_extinction_method SHELXL

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'Fc^*^=kFc[1+0.001xFc^2^\l^3^/sin(2\q)]^-1/4^'

_refine_ls_abs_structure_details

'Flack H D (1983), Acta Cryst. A39, 876-881'

_refine_ls_abs_structure_Flack 0.31(8)

_refine_ls_number_reflns 9894

_refine_ls_number_parameters 453

_refine_ls_number_restraints 0

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_refine_ls_R_factor_gt 0.0608

_refine_ls_wR_factor_ref 0.1791

_refine_ls_wR_factor_gt 0.1547

_refine_ls_goodness_of_fit_ref 1.049

_refine_ls_restrained_S_all 1.049

_refine_ls_shift/su_max 0.001

_refine_ls_shift/su_mean 0.000

loop_

_atom_site_label

_atom_site_type_symbol

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_atom_site_fract_y

_atom_site_fract_z

_atom_site_U_iso_or_equiv

_atom_site_adp_type

_atom_site_occupancy

_atom_site_symmetry_multiplicity

_atom_site_calc_flag

_atom_site_refinement_flags

_atom_site_disorder_assembly

_atom_site_disorder_group

Pd1 Pd 0.69589(3) 0.89588(3) 0.81254(3) 0.03370(17) Uani 1 1 d . . .

Br1 Br 0.64762(5) 0.97271(4) 0.90497(5) 0.0469(2) Uani 1 1 d . . .

P1 P 0.61750(10) 0.88423(10) 0.73275(11) 0.0338(4) Uani 1 1 d . . .

O1 O 0.7288(3) 0.9696(3) 0.7496(3) 0.0462(15) Uani 1 1 d . . .

C1 C 0.7494(4) 0.9643(4) 0.6819(5) 0.0419(19) Uani 1 1 d . . .

Pd2 Pd 0.75838(3) 0.83879(3) 0.71553(3) 0.03643(18) Uani 1 1 d . . .

Br2 Br 0.83957(5) 0.79654(6) 0.62787(5) 0.0578(3) Uani 1 1 d . . .

P2 P 0.75074(10) 0.75801(10) 0.79247(12) 0.0405(5) Uani 1 1 d . . .

O2 O 0.7584(3) 0.9162(3) 0.6476(3) 0.0498(16) Uani 1 1 d . . .

C2 C 0.7630(7) 1.0207(5) 0.6377(6) 0.075(4) Uani 1 1 d . . .

H2A H 0.7543 1.0544 0.6697 0.113 Uiso 1 1 calc R . .

H2B H 0.7391 1.0222 0.5917 0.113 Uiso 1 1 calc R . .

H2C H 0.8041 1.0212 0.6236 0.113 Uiso 1 1 calc R . .

C3 C 0.8177(5) 0.8966(4) 0.8457(5) 0.047(2) Uani 1 1 d . . .

O3 O 0.7740(3) 0.9019(3) 0.8798(4) 0.0546(17) Uani 1 1 d . . .

O4 O 0.8291(3) 0.8780(4) 0.7781(4) 0.0555(17) Uani 1 1 d . . .

C4 C 0.8756(6) 0.9205(9) 0.8892(8) 0.105(6) Uani 1 1 d . . .

H4A H 0.8653 0.9329 0.9406 0.158 Uiso 1 1 calc R . .

H4B H 0.8918 0.9535 0.8612 0.158 Uiso 1 1 calc R . .

H4C H 0.9046 0.8894 0.8920 0.158 Uiso 1 1 calc R . .

C11 C 0.6321(4) 0.8251(4) 0.6662(5) 0.0357(18) Uani 1 1 d . . .

C12 C 0.6888(4) 0.8046(4) 0.6543(4) 0.0350(17) Uani 1 1 d . . .

C13 C 0.5957(5) 0.7614(5) 0.5638(5) 0.051(2) Uani 1 1 d . . .

H13 H 0.5648 0.7467 0.5339 0.062 Uiso 1 1 calc R . .

C14 C 0.6538(5) 0.7433(5) 0.5512(5) 0.050(2) Uani 1 1 d . . .

H14 H 0.6617 0.7160 0.5122 0.060 Uiso 1 1 calc R . .

C15 C 0.7004(4) 0.7652(4) 0.5960(5) 0.045(2) Uani 1 1 d . . .

H15 H 0.7391 0.7530 0.5864 0.054 Uiso 1 1 calc R . .

C16 C 0.5855(4) 0.8022(5) 0.6223(5) 0.045(2) Uani 1 1 d . . .

H16 H 0.5469 0.8145 0.6325 0.054 Uiso 1 1 calc R . .

C21 C 0.5474(4) 0.8658(4) 0.7809(5) 0.0409(19) Uani 1 1 d . . .

C22 C 0.5316(4) 0.8085(5) 0.7939(5) 0.048(2) Uani 1 1 d . . .

H22 H 0.5558 0.7782 0.7758 0.057 Uiso 1 1 calc R . .

C23 C 0.4796(5) 0.7944(5) 0.8339(6) 0.059(3) Uani 1 1 d . . .

H23 H 0.4681 0.7551 0.8408 0.070 Uiso 1 1 calc R . .

C24 C 0.4463(5) 0.8395(6) 0.8625(7) 0.068(3) Uani 1 1 d . . .

H24 H 0.4119 0.8308 0.8898 0.081 Uiso 1 1 calc R . .

C25 C 0.4621(5) 0.8978(8) 0.8522(6) 0.072(4) Uani 1 1 d . . .

H25 H 0.4392 0.9280 0.8735 0.086 Uiso 1 1 calc R . .

C26 C 0.5124(4) 0.9112(5) 0.8100(5) 0.049(2) Uani 1 1 d . . .

H26 H 0.5228 0.9506 0.8011 0.059 Uiso 1 1 calc R . .

C31 C 0.6038(4) 0.9487(4) 0.6727(5) 0.0415(19) Uani 1 1 d . . .

C32 C 0.6036(5) 1.0069(4) 0.7044(5) 0.052(2) Uani 1 1 d . . .

H32 H 0.6120 1.0122 0.7566 0.062 Uiso 1 1 calc R . .

C33 C 0.5915(5) 1.0538(5) 0.6603(6) 0.060(3) Uani 1 1 d . . .

H33 H 0.5921 1.0916 0.6819 0.072 Uiso 1 1 calc R . .

C34 C 0.5780(6) 1.0469(5) 0.5812(7) 0.069(3) Uani 1 1 d . . .

H34 H 0.5689 1.0796 0.5507 0.083 Uiso 1 1 calc R . .

C35 C 0.5784(7) 0.9918(5) 0.5505(6) 0.067(3) Uani 1 1 d . . .

H35 H 0.5697 0.9869 0.4983 0.080 Uiso 1 1 calc R . .

C36 C 0.5913(5) 0.9429(5) 0.5944(6) 0.056(3) Uani 1 1 d . . .

H36 H 0.5917 0.9056 0.5715 0.068 Uiso 1 1 calc R . .

C41 C 0.6892(4) 0.7670(4) 0.8570(5) 0.0404(19) Uani 1 1 d . . .

C42 C 0.6662(4) 0.8236(4) 0.8713(4) 0.0368(18) Uani 1 1 d . . .

C43 C 0.6221(4) 0.8290(4) 0.9283(5) 0.042(2) Uani 1 1 d . . .

H43 H 0.6056 0.8661 0.9379 0.051 Uiso 1 1 calc R . .

C44 C 0.6029(5) 0.7817(5) 0.9696(5) 0.052(2) Uani 1 1 d . . .

H44 H 0.5744 0.7873 1.0078 0.062 Uiso 1 1 calc R . .

C45 C 0.6242(5) 0.7265(5) 0.9566(6) 0.056(3) Uani 1 1 d . . .

H45 H 0.6098 0.6942 0.9843 0.067 Uiso 1 1 calc R . .

C46 C 0.6685(5) 0.7193(4) 0.9002(6) 0.053(2) Uani 1 1 d . . .

H46 H 0.6843 0.6818 0.8916 0.064 Uiso 1 1 calc R . .

C51 C 0.7406(5) 0.6890(4) 0.7432(5) 0.049(2) Uani 1 1 d . . .

C52 C 0.7885(5) 0.6568(4) 0.7199(5) 0.056(2) Uani 1 1 d . . .

H52 H 0.8266 0.6701 0.7310 0.067 Uiso 1 1 calc R . .

C53 C 0.7803(8) 0.6037(5) 0.6791(7) 0.077(4) Uani 1 1 d . . .

H53 H 0.8130 0.5820 0.6625 0.092 Uiso 1 1 calc R . .

C54 C 0.7224(8) 0.5831(5) 0.6633(7) 0.076(4) Uani 1 1 d . . .

H54 H 0.7171 0.5472 0.6377 0.091 Uiso 1 1 calc R . .

C55 C 0.6750(7) 0.6149(6) 0.6850(7) 0.073(3) Uani 1 1 d . . .

H55 H 0.6371 0.6015 0.6731 0.088 Uiso 1 1 calc R . .

C56 C 0.6823(6) 0.6680(5) 0.7253(6) 0.062(3) Uani 1 1 d . . .

H56 H 0.6493 0.6897 0.7405 0.074 Uiso 1 1 calc R . .

C61 C 0.8151(5) 0.7472(5) 0.8539(5) 0.054(3) Uani 1 1 d . . .

C62 C 0.8078(5) 0.7264(5) 0.9285(5) 0.060(3) Uani 1 1 d . . .

H62 H 0.7702 0.7204 0.9490 0.072 Uiso 1 1 calc R . .

C63 C 0.8590(7) 0.7146(6) 0.9728(6) 0.077(4) Uani 1 1 d . . .

H63 H 0.8544 0.7007 1.0231 0.092 Uiso 1 1 calc R . .

C64 C 0.9143(6) 0.7226(7) 0.9453(7) 0.074(4) Uani 1 1 d . . .

H64 H 0.9470 0.7126 0.9754 0.089 Uiso 1 1 calc R . .

C65 C 0.9220(5) 0.7460(7) 0.8713(7) 0.079(4) Uani 1 1 d . . .

H65 H 0.9598 0.7537 0.8526 0.095 Uiso 1 1 calc R . .

C66 C 0.8721(5) 0.7578(7) 0.8252(6) 0.067(3) Uani 1 1 d . . .

H66 H 0.8770 0.7727 0.7754 0.080 Uiso 1 1 calc R . .

loop_

_atom_site_aniso_label

_atom_site_aniso_U_11

_atom_site_aniso_U_22

_atom_site_aniso_U_33

_atom_site_aniso_U_23

_atom_site_aniso_U_13

_atom_site_aniso_U_12

Pd1 0.0365(3) 0.0355(3) 0.0291(3) 0.0011(2) -0.0005(2) -0.0004(3)

Br1 0.0587(6) 0.0422(5) 0.0399(4) -0.0022(3) 0.0016(4) 0.0028(4)

P1 0.0342(11) 0.0350(11) 0.0324(9) -0.0003(8) -0.0009(8) 0.0030(9)

O1 0.057(4) 0.039(3) 0.043(3) 0.002(2) 0.004(3) -0.008(3)

C1 0.043(5) 0.041(5) 0.042(4) -0.005(4) -0.004(4) 0.000(4)

Pd2 0.0337(3) 0.0426(4) 0.0329(3) 0.0015(2) -0.0001(2) 0.0040(3)

Br2 0.0482(6) 0.0744(7) 0.0508(5) 0.0028(5) 0.0076(4) 0.0159(5)

P2 0.0412(12) 0.0408(12) 0.0395(10) 0.0009(8) -0.0013(9) 0.0105(10)

O2 0.066(5) 0.046(4) 0.037(3) 0.004(3) 0.002(3) -0.008(3)

C2 0.120(11) 0.050(7) 0.056(6) 0.009(5) 0.024(6) -0.011(7)

C3 0.068(7) 0.047(5) 0.026(4) 0.001(4) 0.005(4) 0.001(5)

O3 0.054(4) 0.063(5) 0.047(3) 0.007(3) -0.016(3) -0.008(4)

O4 0.032(3) 0.076(5) 0.058(4) -0.007(3) 0.000(3) -0.008(3)

C4 0.047(7) 0.185(19) 0.084(9) -0.046(10) -0.015(6) -0.005(9)

C11 0.037(5) 0.032(4) 0.038(4) -0.004(3) 0.002(3) 0.000(3)

C12 0.030(4) 0.043(5) 0.032(3) 0.004(3) -0.002(3) 0.007(4)

C13 0.061(7) 0.058(6) 0.035(4) -0.007(4) -0.010(4) -0.010(5)

C14 0.059(6) 0.050(6) 0.041(4) -0.006(4) -0.005(4) 0.007(5)

C15 0.039(5) 0.055(6) 0.041(4) -0.010(4) -0.003(4) 0.010(4)

C16 0.029(4) 0.052(6) 0.056(5) -0.003(4) -0.001(4) 0.007(4)

C21 0.036(4) 0.055(5) 0.032(4) 0.000(4) -0.005(3) 0.001(4)

C22 0.041(5) 0.050(6) 0.051(5) -0.002(4) 0.003(4) -0.003(4)

C23 0.055(6) 0.063(7) 0.058(6) 0.009(5) -0.001(5) -0.008(6)

C24 0.043(6) 0.085(9) 0.075(7) 0.004(6) 0.005(5) -0.009(6)

C25 0.037(6) 0.124(12) 0.054(6) -0.011(6) 0.003(4) 0.012(7)

C26 0.030(4) 0.064(6) 0.052(5) -0.009(4) 0.002(4) 0.009(4)

C31 0.051(5) 0.035(4) 0.039(4) 0.004(3) 0.006(4) 0.006(4)

C32 0.070(7) 0.045(5) 0.040(5) -0.004(4) -0.009(4) 0.004(5)

C33 0.078(8) 0.045(6) 0.057(6) -0.006(5) 0.000(5) 0.004(6)

C34 0.087(9) 0.061(7) 0.060(6) 0.023(5) 0.001(6) 0.012(6)

C35 0.106(10) 0.053(7) 0.041(5) 0.004(4) -0.003(5) 0.013(7)

C36 0.077(8) 0.049(6) 0.044(5) 0.003(4) 0.004(5) 0.011(5)

C41 0.044(5) 0.033(4) 0.044(4) 0.006(3) -0.002(4) 0.002(4)

C42 0.040(5) 0.044(5) 0.027(3) 0.007(3) 0.004(3) 0.000(4)

C43 0.046(5) 0.044(5) 0.037(4) -0.003(3) 0.005(3) 0.003(4)

C44 0.059(6) 0.054(6) 0.042(5) 0.009(4) 0.013(4) 0.005(5)

C45 0.058(6) 0.051(6) 0.058(5) 0.021(5) 0.005(5) -0.006(5)

C46 0.064(7) 0.035(5) 0.060(6) 0.004(4) 0.004(5) 0.004(5)

C51 0.065(7) 0.038(5) 0.045(5) 0.006(4) -0.001(4) 0.006(5)

C52 0.072(7) 0.043(5) 0.051(5) 0.002(4) 0.006(5) 0.016(5)

C53 0.117(12) 0.045(6) 0.068(7) 0.002(5) 0.018(7) 0.023(7)

C54 0.126(13) 0.038(6) 0.064(7) 0.002(5) -0.010(7) 0.012(7)

C55 0.091(9) 0.064(8) 0.064(7) 0.003(6) -0.019(6) -0.012(7)

C56 0.070(7) 0.053(6) 0.062(6) -0.009(5) -0.013(5) 0.014(5)

C61 0.051(6) 0.066(7) 0.044(5) -0.002(4) -0.008(4) 0.020(5)

C62 0.063(7) 0.073(7) 0.043(5) 0.004(4) -0.009(5) 0.017(6)

C63 0.103(11) 0.090(10) 0.039(5) 0.013(5) -0.017(6) 0.010(8)

C64 0.057(7) 0.099(10) 0.067(7) 0.006(6) -0.022(6) 0.020(7)

C65 0.037(6) 0.118(12) 0.083(8) 0.017(8) -0.012(5) 0.026(7)

C66 0.041(6) 0.109(10) 0.051(6) 0.007(6) -0.003(4) 0.020(6)

_geom_special_details

;

All esds (except the esd in the dihedral angle between two l.s. planes)

are estimated using the full covariance matrix. The cell esds are taken

into account individually in the estimation of esds in distances, angles

and torsion angles; correlations between esds in cell parameters are only

used when they are defined by crystal symmetry. An approximate (isotropic)

treatment of cell esds is used for estimating esds involving l.s. planes.

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loop_

_geom_bond_atom_site_label_1

_geom_bond_atom_site_label_2

_geom_bond_distance

_geom_bond_site_symmetry_2

_geom_bond_publ_flag

Pd1 C42 2.035(8) . ?

Pd1 O3 2.115(7) . ?

Pd1 O1 2.122(6) . ?

Pd1 P1 2.258(2) . ?

Pd1 Pd2 2.5411(9) . ?

Pd1 Br1 2.5967(11) . ?

P1 C11 1.792(8) . ?

P1 C31 1.814(9) . ?

P1 C21 1.835(9) . ?

O1 C1 1.265(11) . ?

C1 O2 1.254(11) . ?

C1 C2 1.516(13) . ?

Pd2 C12 2.045(8) . ?

Pd2 O2 2.104(6) . ?

Pd2 O4 2.122(7) . ?

Pd2 P2 2.263(2) . ?

Pd2 Br2 2.5613(12) . ?

P2 C51 1.790(10) . ?

P2 C41 1.792(9) . ?

P2 C61 1.815(10) . ?

C3 O3 1.156(12) . ?

C3 O4 1.269(11) . ?

C3 C4 1.601(16) . ?

C11 C12 1.376(11) . ?

C11 C16 1.396(12) . ?

C12 C15 1.368(12) . ?

C13 C16 1.387(14) . ?

C13 C14 1.389(15) . ?

C14 C15 1.397(14) . ?

C21 C22 1.360(14) . ?

C21 C26 1.387(13) . ?

C22 C23 1.398(14) . ?

C23 C24 1.359(17) . ?

C24 C25 1.37(2) . ?

C25 C26 1.383(15) . ?

C31 C36 1.389(13) . ?

C31 C32 1.422(13) . ?

C32 C33 1.333(15) . ?

C33 C34 1.409(16) . ?

C34 C35 1.352(17) . ?

C35 C36 1.369(15) . ?

C41 C46 1.390(13) . ?

C41 C42 1.400(12) . ?

C42 C43 1.405(12) . ?

C43 C44 1.356(14) . ?

C44 C45 1.355(16) . ?

C45 C46 1.405(15) . ?

C51 C52 1.363(15) . ?

C51 C56 1.432(16) . ?

C52 C53 1.403(17) . ?

C53 C54 1.41(2) . ?

C54 C55 1.34(2) . ?

C55 C56 1.395(16) . ?

C61 C62 1.382(14) . ?

C61 C66 1.399(16) . ?

C62 C63 1.411(16) . ?

C63 C64 1.35(2) . ?

C64 C65 1.393(18) . ?

C65 C66 1.404(14) . ?

loop_

_geom_angle_atom_site_label_1

_geom_angle_atom_site_label_2

_geom_angle_atom_site_label_3

_geom_angle

_geom_angle_site_symmetry_1

_geom_angle_site_symmetry_3

_geom_angle_publ_flag

C42 Pd1 O3 92.9(3) . . ?

C42 Pd1 O1 178.3(3) . . ?

O3 Pd1 O1 86.5(3) . . ?

C42 Pd1 P1 87.3(2) . . ?

O3 Pd1 P1 174.6(2) . . ?

O1 Pd1 P1 93.04(19) . . ?

C42 Pd1 Pd2 96.1(2) . . ?

O3 Pd1 Pd2 86.1(2) . . ?

O1 Pd1 Pd2 82.27(18) . . ?

P1 Pd1 Pd2 88.45(6) . . ?

C42 Pd1 Br1 95.2(2) . . ?

O3 Pd1 Br1 88.2(2) . . ?

O1 Pd1 Br1 86.46(18) . . ?

P1 Pd1 Br1 97.17(6) . . ?

Pd2 Pd1 Br1 167.67(4) . . ?

C11 P1 C31 105.2(4) . . ?

C11 P1 C21 106.3(4) . . ?

C31 P1 C21 107.1(4) . . ?

C11 P1 Pd1 109.6(3) . . ?

C31 P1 Pd1 112.9(3) . . ?

C21 P1 Pd1 115.1(3) . . ?

C1 O1 Pd1 121.8(6) . . ?

O2 C1 O1 125.4(8) . . ?

O2 C1 C2 117.2(8) . . ?

O1 C1 C2 117.4(8) . . ?

C12 Pd2 O2 91.4(3) . . ?

C12 Pd2 O4 177.5(3) . . ?

O2 Pd2 O4 86.5(3) . . ?

C12 Pd2 P2 86.6(2) . . ?

O2 Pd2 P2 175.1(2) . . ?

O4 Pd2 P2 95.4(2) . . ?

C12 Pd2 Pd1 96.1(2) . . ?

O2 Pd2 Pd1 86.99(19) . . ?

O4 Pd2 Pd1 82.54(19) . . ?

P2 Pd2 Pd1 88.77(6) . . ?

C12 Pd2 Br2 95.9(2) . . ?

O2 Pd2 Br2 88.8(2) . . ?

O4 Pd2 Br2 85.31(19) . . ?

P2 Pd2 Br2 95.86(7) . . ?

Pd1 Pd2 Br2 167.35(4) . . ?

C51 P2 C41 107.2(5) . . ?

C51 P2 C61 105.3(5) . . ?

C41 P2 C61 105.7(4) . . ?

C51 P2 Pd2 115.5(3) . . ?

C41 P2 Pd2 109.5(3) . . ?

C61 P2 Pd2 113.0(4) . . ?

C1 O2 Pd2 117.1(5) . . ?

O3 C3 O4 132.6(10) . . ?

O3 C3 C4 115.1(9) . . ?

O4 C3 C4 112.2(9) . . ?

C3 O3 Pd1 115.2(6) . . ?

C3 O4 Pd2 117.0(7) . . ?

C12 C11 C16 119.6(8) . . ?

C12 C11 P1 121.2(7) . . ?

C16 C11 P1 119.1(7) . . ?

C15 C12 C11 120.5(8) . . ?

C15 C12 Pd2 118.6(6) . . ?

C11 C12 Pd2 120.6(6) . . ?

C16 C13 C14 117.8(9) . . ?

C13 C14 C15 121.3(9) . . ?

C12 C15 C14 119.6(8) . . ?

C13 C16 C11 121.1(8) . . ?

C22 C21 C26 119.6(9) . . ?

C22 C21 P1 121.1(7) . . ?

C26 C21 P1 119.2(8) . . ?

C21 C22 C23 121.3(10) . . ?

C24 C23 C22 118.1(11) . . ?

C23 C24 C25 121.9(11) . . ?

C24 C25 C26 119.4(12) . . ?

C25 C26 C21 119.7(11) . . ?

C36 C31 C32 117.5(8) . . ?

C36 C31 P1 121.1(7) . . ?

C32 C31 P1 121.4(7) . . ?

C33 C32 C31 120.9(9) . . ?

C32 C33 C34 120.7(10) . . ?

C35 C34 C33 118.7(10) . . ?

C34 C35 C36 121.7(10) . . ?

C35 C36 C31 120.5(10) . . ?

C46 C41 C42 119.1(8) . . ?

C46 C41 P2 120.5(7) . . ?

C42 C41 P2 120.0(7) . . ?

C41 C42 C43 117.8(8) . . ?

C41 C42 Pd1 121.4(6) . . ?

C43 C42 Pd1 120.8(7) . . ?

C44 C43 C42 121.7(9) . . ?

C45 C44 C43 121.6(9) . . ?

C44 C45 C46 118.2(9) . . ?

C41 C46 C45 121.5(9) . . ?

C52 C51 C56 119.3(10) . . ?

C52 C51 P2 120.2(9) . . ?

C56 C51 P2 120.5(8) . . ?

C51 C52 C53 119.9(13) . . ?

C52 C53 C54 120.0(13) . . ?

C55 C54 C53 120.5(12) . . ?

C54 C55 C56 120.3(13) . . ?

C55 C56 C51 120.0(11) . . ?

C62 C61 C66 119.9(10) . . ?

C62 C61 P2 119.7(9) . . ?

C66 C61 P2 120.4(7) . . ?

C61 C62 C63 118.2(11) . . ?

C64 C63 C62 122.8(10) . . ?

C63 C64 C65 119.2(10) . . ?

C64 C65 C66 119.6(12) . . ?

C61 C66 C65 120.2(10) . . ?

_diffrn_measured_fraction_theta_max 0.989

_diffrn_reflns_theta_full 27.48

_diffrn_measured_fraction_theta_full 0.989

_refine_diff_density_max 1.680

_refine_diff_density_min -1.505

_refine_diff_density_rms 0.414

Synthesis of 3: **4b** (50 mg, 0.028 mmol) was dissolved in dichloromethane (6 mL) and silver tetrafluoroborate (24 mg, 0.123 mmol) in acetonitrile (1.5 mL) was added under vigorous stirring. The stirring was continued for 1 hour in the absence of light. The pale yellow solution was filtered to remove precipitated silver bromide and dried under vacuum to give a yellow oil. This crude product exhibited a single signal at 21.5 ppm in ^{31}P-NMR spectroscopy. It was dissolved in the minimum amount of acetonitrile and the addition of diethyl ether precipitated a pale yellow solid, which was collected by filtration and washed with diethyl ether (49 mg). Its ^{31}P-NMR spectrum showed two broad signals at 21.5 ppm and 23.8 ppm but when one drop of acetonitrile was added to the sample the spectrum changed again to a single sharp signal at 21.5 ppm.

Synthesis of 4c: A sample of **3**, prepared as described above, starting from 50 mg (0.028 mmol) of **4b**, was dissolved in acetone (3 mL) and a solution of tetrabutylammonium iodide (62 mg, 0.168 mmol) in acetone (3 mL) was added. A fine orange precipitate immediately formed, which was collected by filtration and washed with acetone and diethyl ether (51 mg, 93 %).

^{31}P{^{1}H} NMR ($CDCl_3$, 20 °C): 24.0 (s). ^{1}H NMR ($CDCl_3$, 20 °C): 7.52-7.41 (m, 12H), 7.28-7.10 (m, 32H), 6.51-6.41 (m, 12H). Anal. calcd. for $C_{72}H_{56}I_4P_4Pd_4$: C 43.08, H 2.98. Found: C 43.71, H 2.85.

Typical catalytic diboration and subsequent oxidation: Bis(catecholato)diboron (0.6 mmol) was added to a solution of the catalyst (5 mol%, 0.01 mmol Pd) and sodium acetate (0.2 mmol) in tetrahydrofurane (2 mL) under nitrogen. The solution was stirred for 5 minutes and the substrate (0.2 mmol) was then added. The mixture was stirred for 4 hours at room temperature. NaOH(aq) (3 M, 1 mL) and H_2O_2 (30 %, 1 mL) were added carefully and stirring at room temperature was continued for 2 hours. The oxidation was quenched by adding a saturated aqueous solution of sodium thiosulfate (1 mL) and NaOH(aq) (1 M, 10 mL). Then the reaction mixture was extracted with ethyl acetate (3 x 20 mL) and the united organic phases were washed with brine (20 mL), dried over magnesium sulfate and carefully dried *in vacuo* so that the styrene was not removed. The products obtained were analyzed by ^{1}H NMR spectroscopy to determine the degree of conversion and the nature of the reaction products.

Typical catalytic diboration, subsequent Suzuki reaction and oxidation: Bis(catecholato)diboron (0.6 mmol) was added to a solution of the catalyst (5 mol%, 0.01 mmol Pd) and NaOAc (0.2 mmol) in tetrahydrofurane (2 mL) under nitrogen. The solution was stirred for 5 minutes, the substrate (0.2 mmol) was added and the stirring was continued for 4 hours at room temperature. After heating to reflux, cesium carbonate (0.6 mmol), substrate (0.4 mmol) and water (degassed, 0.2 mL) were added and the reaction mixture was stirred for 15 hours. After cooling to room temperature, NaOH(aq) (3 M, 1 mL) and H_2O_2 (30 %, 1 mL) were added carefully and stirring was continued for 2 hours. The oxidation was quenched by adding a saturated aqueous solution of sodium thiosulfate (1 mL) and NaOH(aq) (1 M, 10 mL). Then the reaction mixture was extracted with ethyl acetate (3 x 20 mL) and the united organic phases were washed with brine (20 mL), dried over magnesium sulfate and dried *in vacuo*. The products obtained were analyzed by ^{1}H NMR spectroscopy[1] to determine the degree of conversion and the nature of the reaction products.

(±)-1-Cyclohexyl-2-phenylethanol: ^{1}H NMR (400 MHz, $CDCl_3$) d 7.23 (1H, m), 6.78 (3H, m), 3.80 (3H, 1s, OMe), 3.58 (1H, ddd, *J* = 9.6, 6.0, 3.6 Hz), 2.85 (1H, dd, *J* = 13.6, 3.6Hz), 2.56 (1H, dd, *J* = 13.6, 9.5 Hz), 2.04-1.67 (5H, m), 1.44-1.41 (1H, m), 1.29-1.07 (5H, m) ppm; ^{13}C NMR (75 MHz, $CDCl_3$) d 159.9, 141.0, 129.7, 122.8, 115.9, 11.9, 77.0, 55.3, 43.4, 41.0, 29.5, 28.2, 26.7, 26.4 ppm.

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1. S. P. Miller, J. B. Morgan, F. J. Nepveux, J. P. Morken, *Org. Lett.*, **2004**, *6*, 131.

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