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Dichlorido(4,5-diazafluoren-9-one-κ^2N,N')palladium(II)

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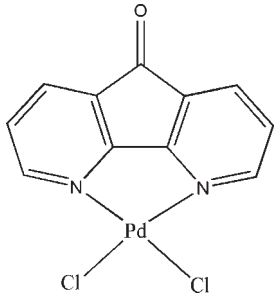
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.029; wR factor = 0.058; data-to-parameter ratio = 15.2.

The structure of the title compound, $[PdCl_2(C_{11}H_6N_2O)]$, shows a nearly square-planar geometry for the Pd^{II} atom within a Cl_2N_2 donor set.

Related literature

For related palladium complexes, see: Klein *et al.* (1998).



Experimental

Crystal data

$[PdCl_2(C_{11}H_6N_2O)]$ M_r = 359.48
Monoclinic, $P2_1/c$
a = 5.131 (5) Å
b = 17.105 (5) Å
c = 12.763 (5) Å
β = 99.183 (5)°
V = 1105.8 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.14 mm^{-1}
T = 293 K
0.35 × 0.33 × 0.25 mm

Data collection

Bruker SMART APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.477, T_{max} = 0.586
6785 measured reflections
2703 independent reflections
2198 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.058
S = 1.04
2703 reflections
178 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.54 e Å^{-3}

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2532).

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supporting information

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S1. Comment

The title compound, (I), Fig. 1, shows the palladium atom to be coordinated by two chlorides and two nitrogen atoms, the latter derived from a chelating 4,5-diazafluoren-9-one (dafo) ligand. The coordination geometry is based on a square planar arrangement of the four donor atoms. Related palladium structures featuring dafo ligands are known (Klein *et al.*, 1998).

S2. Experimental

Compound (I) was synthesized hydrothermally from an aqueous mixture (10.0 ml) containing $PdCl_2$ (0.1758 g) and 4,5-diazafluoren-9-one (0.301 g) in a 30 ml Teflon-lined stainless steel vessel. In the vessel, the solution was heated to 413 K for 72 h then cooled to room temperature at a rate of 10 K per hour. Brown prisms were obtained after washed thoroughly with deionized water and air-drying.

S3. Refinement

All H atoms were refined: range of C-H = 0.89 (3) to 0.99 (4) Å.

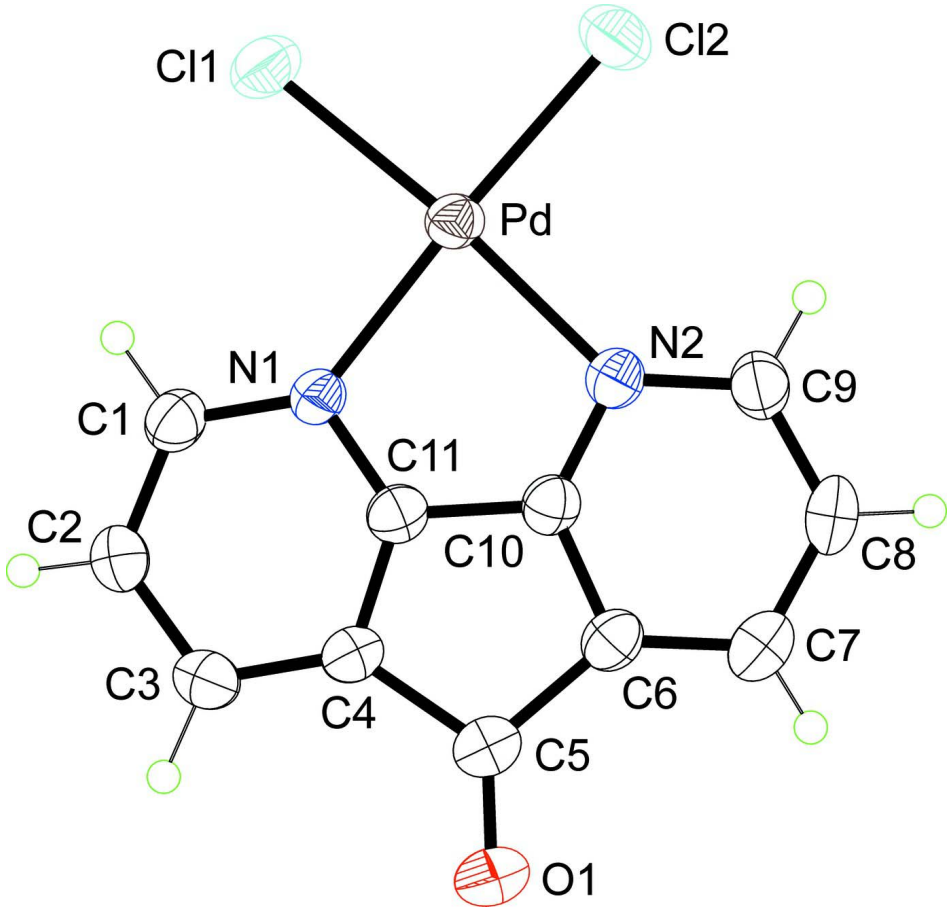

Figure 1

Molecular structure of (I) with displacement ellipsoids drawn at the 50% probability level.

Dichlorido(4,5-diazafluoren-9-one-κ^2N,N')palladium(II)

Crystal data

$[PdCl_2(C_{11}H_6N_2O)]$
$M_r = 359.48$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 5.131\ (5)$ Å
$b = 17.105\ (5)$ Å
$c = 12.763\ (5)$ Å
$\beta = 99.183\ (5)°$
$V = 1105.8\ (12)$ Å^3
$Z = 4$
$F(000) = 696$
$D_x = 2.159$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å
Cell parameters from 13853 reflections
$\theta = 1.8–28.2°$
$\mu = 2.14$ mm^{-1}
$T = 293$ K
Block, yellow
0.35 × 0.33 × 0.25 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 10 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.477$, $T_{max} = 0.586$
6785 measured reflections
2703 independent reflections
2198 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$

θ_{max} = 28.4°, θ_{min} = 2.0°
h = −6→6
k = −22→13
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.058
S = 1.04
2703 reflections
178 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0199P)^2 + 0.6053P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.54 e Å^{-3}

Special details

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

Refinement. 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 $F^2 > \sigma(F^2)$ is used only for calculating Rfactors(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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
Pd1	0.08571 (4)	0.152850 (13)	0.283179 (18)	0.03075 (8)
N2	0.2819 (5)	0.16090 (14)	0.15176 (19)	0.0336 (6)
N1	0.2865 (4)	0.04745 (13)	0.30833 (19)	0.0303 (5)
C6	0.6231 (5)	0.08217 (18)	0.0892 (2)	0.0343 (7)
C4	0.6221 (6)	−0.01682 (17)	0.2253 (2)	0.0322 (6)
C5	0.7568 (6)	0.00609 (19)	0.1320 (2)	0.0378 (7)
O1	0.9357 (5)	−0.02803 (14)	0.10159 (19)	0.0532 (6)
C11	0.4449 (5)	0.04170 (17)	0.2364 (2)	0.0319 (6)
C10	0.4439 (5)	0.10020 (17)	0.1557 (2)	0.0317 (6)
C3	0.6398 (6)	−0.0784 (2)	0.2962 (3)	0.0402 (8)
C8	0.4677 (7)	0.1976 (2)	−0.0016 (3)	0.0434 (8)
C1	0.3028 (6)	−0.01242 (19)	0.3780 (3)	0.0367 (7)
C9	0.2942 (6)	0.2103 (2)	0.0695 (3)	0.0397 (7)
C7	0.6379 (7)	0.1337 (2)	0.0064 (3)	0.0414 (8)
C2	0.4742 (6)	−0.0747 (2)	0.3724 (3)	0.0413 (8)
Cl1	−0.10542 (16)	0.13410 (5)	0.42956 (7)	0.0445 (2)
Cl2	−0.11747 (16)	0.26981 (5)	0.24949 (7)	0.0479 (2)
H1	0.187 (6)	−0.0098 (17)	0.429 (2)	0.038 (9)*
H3	0.762 (7)	−0.123 (2)	0.295 (3)	0.052 (10)*
H4	0.753 (6)	0.1289 (19)	−0.038 (3)	0.048 (10)*
H2	0.477 (6)	−0.115 (2)	0.420 (3)	0.046 (9)*
H5	0.465 (6)	0.2338 (19)	−0.055 (3)	0.043 (9)*
H6	0.181 (6)	0.2543 (19)	0.064 (2)	0.046 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd1	0.02879 (12)	0.02863 (13)	0.03619 (14)	−0.00123 (9)	0.00940 (9)	−0.00117 (10)
N2	0.0321 (13)	0.0332 (14)	0.0364 (14)	−0.0019 (11)	0.0084 (10)	0.0016 (11)
N1	0.0293 (12)	0.0303 (13)	0.0328 (14)	−0.0010 (10)	0.0097 (10)	0.0015 (10)
C6	0.0300 (15)	0.0392 (18)	0.0341 (17)	−0.0042 (13)	0.0070 (12)	−0.0008 (13)
C4	0.0306 (15)	0.0341 (16)	0.0330 (17)	−0.0017 (12)	0.0086 (12)	−0.0034 (13)
C5	0.0351 (16)	0.0419 (18)	0.0373 (18)	−0.0013 (14)	0.0093 (14)	−0.0042 (14)
O1	0.0540 (14)	0.0596 (16)	0.0516 (15)	0.0154 (12)	0.0258 (12)	0.0029 (12)
C11	0.0281 (15)	0.0337 (16)	0.0340 (17)	−0.0032 (12)	0.0056 (12)	−0.0019 (12)
C10	0.0279 (14)	0.0343 (17)	0.0329 (16)	−0.0027 (12)	0.0049 (12)	0.0010 (13)
C3	0.0390 (18)	0.0366 (18)	0.046 (2)	0.0071 (14)	0.0092 (15)	0.0012 (15)
C8	0.051 (2)	0.045 (2)	0.0340 (19)	−0.0075 (16)	0.0075 (15)	0.0118 (15)
C1	0.0366 (17)	0.0387 (18)	0.0366 (18)	−0.0027 (14)	0.0112 (14)	0.0016 (14)
C9	0.0401 (18)	0.0362 (18)	0.043 (2)	0.0004 (15)	0.0070 (14)	0.0079 (14)
C7	0.0427 (19)	0.049 (2)	0.0345 (19)	−0.0075 (15)	0.0114 (15)	−0.0019 (15)
C2	0.0461 (19)	0.0348 (18)	0.044 (2)	0.0026 (15)	0.0122 (15)	0.0084 (15)
Cl1	0.0490 (5)	0.0435 (5)	0.0461 (5)	−0.0033 (4)	0.0235 (4)	−0.0035 (4)
Cl2	0.0470 (5)	0.0334 (4)	0.0651 (6)	0.0065 (4)	0.0147 (4)	0.0033 (4)

Geometric parameters (Å, º)

Pd1—N1	2.076 (2)	C4—C5	1.521 (4)
Pd1—N2	2.094 (3)	C5—O1	1.203 (4)
Pd1—Cl2	2.2652 (11)	C11—C10	1.436 (4)
Pd1—Cl1	2.2672 (12)	C3—C2	1.391 (5)
N2—C10	1.326 (4)	C8—C9	1.387 (5)
N2—C9	1.356 (4)	C8—C7	1.392 (5)
N1—C11	1.323 (4)	C1—C2	1.390 (5)
N1—C1	1.350 (4)	H3—C3	0.99 (4)
C6—C10	1.382 (4)	H1—C1	0.95 (3)
C6—C7	1.387 (4)	H2—C2	0.92 (4)
C6—C5	1.531 (4)	H5—C8	0.92 (4)
C4—C11	1.375 (4)	H6—C9	0.95 (3)
C4—C3	1.382 (4)	H4—C7	0.89 (3)
N1—Pd1—N2	83.74 (10)	C4—C11—C10	111.1 (3)
N1—Pd1—Cl2	176.78 (7)	N2—C10—C6	128.8 (3)
N2—Pd1—Cl2	93.17 (8)	N2—C10—C11	120.2 (3)
N1—Pd1—Cl1	91.09 (7)	C6—C10—C11	111.0 (3)
N2—Pd1—Cl1	174.80 (7)	C4—C3—C2	116.1 (3)
Cl2—Pd1—Cl1	92.00 (4)	C9—C8—C7	122.3 (3)
C10—N2—C9	114.2 (3)	N1—C1—C2	121.3 (3)
C10—N2—Pd1	107.26 (19)	N2—C9—C8	121.6 (3)
C9—N2—Pd1	138.5 (2)	C6—C7—C8	116.4 (3)
C11—N1—C1	114.8 (3)	C1—C2—C3	122.2 (3)
C11—N1—Pd1	107.58 (19)	C7—C8—H5	121 (2)

C1—N1—Pd1	137.5 (2)	C9—C8—H5	116 (2)
C10—C6—C7	116.7 (3)	N1—C1—H1	115.8 (18)
C10—C6—C5	106.0 (3)	C2—C1—H1	122.8 (18)
C7—C6—C5	137.4 (3)	C1—C2—H2	119 (2)
C11—C4—C3	117.6 (3)	C3—C2—H2	119 (2)
C11—C4—C5	106.5 (3)	N2—C9—H6	116.7 (17)
C3—C4—C5	135.9 (3)	C8—C9—H6	121.8 (17)
O1—C5—C4	126.4 (3)	C4—C3—H3	124 (2)
O1—C5—C6	128.2 (3)	C2—C3—H3	120 (2)
C4—C5—C6	105.3 (2)	C6—C7—H4	124 (2)
N1—C11—C4	128.0 (3)	C8—C7—H4	120 (2)
N1—C11—C10	120.9 (3)		