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trans-Dichlorobis[*N*-(3,5-difluorophenyl)-isopropylideneamine]palladium(II)

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Key indicators

Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.005 Å
R factor = 0.027
wR factor = 0.059
Data-to-parameter ratio = 14.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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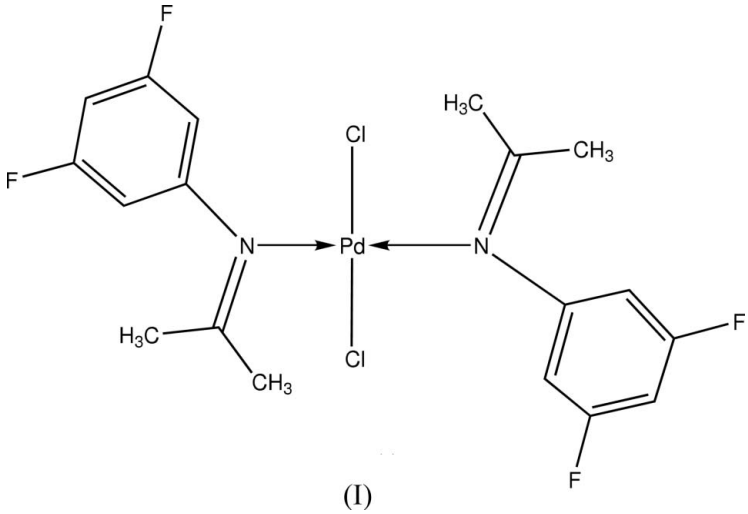
The title compound, $[PdCl_2(C_9H_9F_2N)_2]$, was obtained from the condensation reaction of acetone with *trans*-dichlorobis(3,5-difluoroaniline)palladium(II) under crystallization conditions. The Pd atom lies on an inversion centre and shows a square-planar geometry. The molecules are linked *via* non-classical C—H···Cl and C—H···F interactions.

Comment

Palladium complexes have acquired tremendous importance in recent years due to the main role that palladium has gained in the realm of metal-mediated organic synthesis (Negishi & De Meijere, 2002), becoming a keystone in processes otherwise difficult to achieve or even impossible without the presence of the metal complex, such as the arylation of olefins (Heck reaction; Beletskaya & Cheprakov, 2000). In fact, similar complexes have been successfully employed in this process allowing fine-tuning of electronic effects (Baldovino-Pantaleón *et al.*, 2006), given the facility of modifying the number and disposition of the F atoms in the aromatic ring of the aniline. In this paper, we report the crystal structure of the title compound, (I), as one of a series of compounds we have studied in the course of our research into the synthesis of transition metal complexes bearing fluorinated ligands (Redón *et al.*, 2003, 2002, 2001; Arroyo *et al.*, 2003; Morales-Morales *et al.*, 2001; García *et al.*, 1993, Herrera-Alvarez *et al.*, 2004; Fierro-Arias *et al.*, 2005; Baldovino-Pantaleón *et al.*, 2005).



(I)

Fig. 1 shows the Pd metal centre of (I) to be in a centrosymmetric square-planar environment, with the 3,5-difluorophenylisopropylideneamine and Cl ligands in *trans* positions.

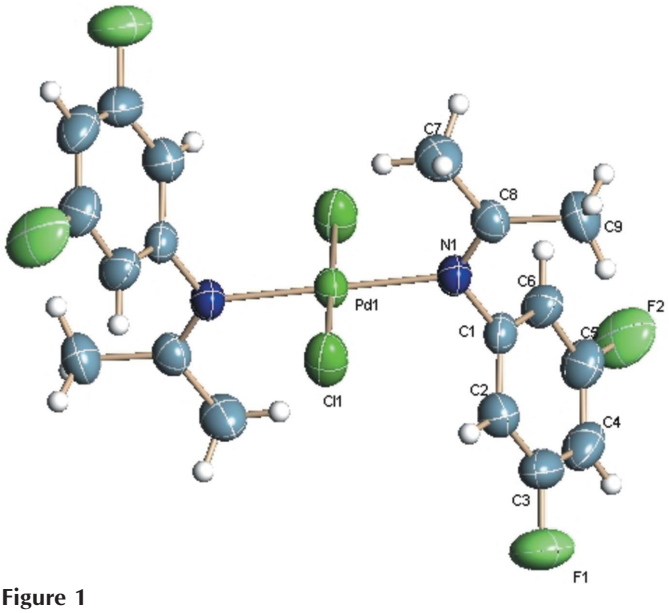


Figure 1
The structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Unlabelled atoms are related to labelled atoms by the symmetry operator $(1-x, 1-y, 1-z)$.

The isopropylideneamine group [r.m.s. deviation 0.0011 (2) Å] makes a dihedral angle of 83.8 (1)° with the plane formed by atoms Pd1/Cl1/N1/Cl1^{i}/N1^{i} [symmetry code: (i) $1-x, 1-y, 1-z$], and the 3,5-difluorophenyl substituent (r.m.s. deviation 0.0054 Å) makes an angle of 73.55 (9)° with the coordination plane. The corresponding angles in dichlorobis(*N*-isopropylideneaniline-N)palladium(II) are 88.1 and 66.8°, respectively (Clegg *et al.*, 1987).

The crystal packing of (I) (Fig. 2) shows non-classical C—H···Cl/F hydrogen bonds (Table 2).

Experimental

Compound (I) was obtained from the condensation reaction of the bis(3,5-difluoroaniline) complex *trans*-[$PdCl_2(C_9H_9F_2N)_2$] (Baldovino-Pantaleón *et al.*, 2006) with acetone, after standing for 4 d under crystallization conditions.

Crystal data

$[PdCl_2(C_9H_9F_2N)_2]$	$Z = 1$
$M_r = 515.64$	$D_x = 1.702$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 7.7654$ (7) Å	Cell parameters from 3473 reflections
$b = 7.9246$ (7) Å	$\theta = 2.5$–$30.7°$
$c = 9.3977$ (8) Å	$\mu = 1.23$ mm^{-1}
$\alpha = 113.735$ (2)°	$T = 291$ (2) K
$\beta = 105.458$ (2)°	Prism, yellow
$\gamma = 92.253$ (2)°	0.12 × 0.10 × 0.08 mm
$V = 503.15$ (8) Å^3	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	1781 independent reflections
	1710 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.029$
Absorption correction: analytical (*SHELXTL*; Sheldrick, 1997*b*)	$\theta_{max} = 25.0°$
	$h = -9 \rightarrow 9$
$T_{min} = 0.867$, $T_{max} = 0.908$	$k = -9 \rightarrow 9$
4178 measured reflections	$l = -11 \rightarrow 11$

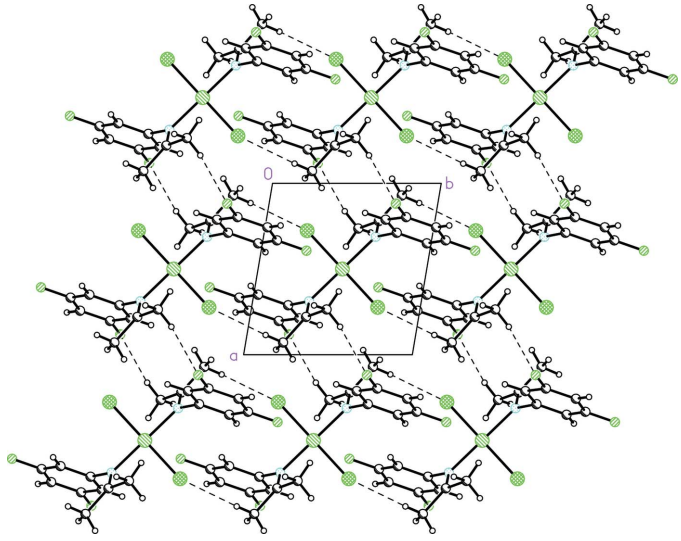


Figure 2
The crystal packing of (I). Dashed lines indicate C—H···Cl/F interactions.

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.027$	$w = 1/[\sigma^2(F_o^2) + (0.0285P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.059$	$(\Delta/\sigma)_{max} = 0.004$
$S = 1.01$	$\Delta\rho_{max} = 0.50$ e Å^{-3}
1781 reflections	$\Delta\rho_{min} = -0.25$ e Å^{-3}
126 parameters	

Table 1
Selected geometric parameters (Å, °).

Pd1—N1	2.0185 (19)	F2—C5	1.349 (3)
Pd1—Cl1	2.2974 (7)	N1—C8	1.285 (3)
F1—C3	1.351 (4)	N1—C1	1.438 (3)
N1—Pd1—Cl1	90.49 (6)	N1—C8—C7	118.6 (2)
C8—N1—C1	121.3 (2)	N1—C8—C9	124.3 (3)
C8—N1—Pd1	124.61 (18)	C7—C8—C9	117.1 (2)
C1—N1—Pd1	114.09 (15)		
Cl1—Pd1—N1—C8	−81.0 (2)	Pd1—N1—C1—C2	−68.5 (3)
Cl1—Pd1—N1—C1	99.82 (17)	Pd1—N1—C8—C7	−5.0 (4)
C8—N1—C1—C6	−70.9 (3)	C1—N1—C8—C9	−6.2 (4)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7B···F1^{i}	0.96	2.54	3.327 (5)	140
C7—H7C···F1^{ii}	0.96	2.47	3.351 (4)	153
C9—H9C···Cl1^{iii}	0.96	2.78	3.694 (3)	161

Symmetry codes: (i) $x, y, z-1$; (ii) $-x, -y+1, -z+1$; (iii) $x, y+1, z$.

H atoms were included in calculated positions, with C—H = 0.96–0.98 Å, and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine

structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL*/*PC* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*/*PC*.

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