Crystal structure of *trans*-[1,3-bis(9-benzyl-9*H*-fluoren-9-yl) benzimidazol-2-ylidene] pyridine palladium(II) dichloride, $C_{52}H_{39}Cl_2N_3Pd$

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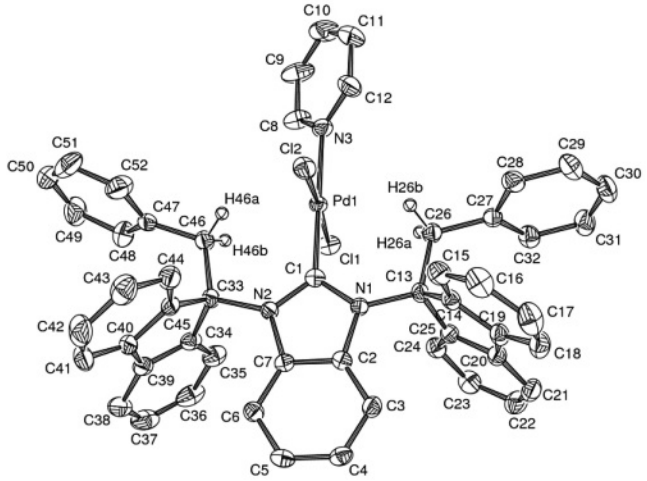

Abstract

$C_{52}H_{39}Cl_2N_3Pd$, tetragonal, $I4_1/a$ (no. 88), a = 40.0310(3) Å, c = 10.4211(1) Å, V = 16699.6 Å^3, Z = 16, $R_{gt}(F)$ = 0.0292, $wR_{ref}(F^2)$ = 0.0709, T = 120 K.

Table 1. Data collection and handling.

Crystal:	yellow blocks, size 0.182×0.192×0.249 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	6.13 cm^{-1}
Diffractometer, scan mode:	CCD Saphire 3 Xcalibur, ω
$2\theta_{max}$:	54°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	68373, 9119
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 6965
$N(param)_{refined}$:	523
Programs:	SIR-97 [4], SHELX [5], PLATON [6]

Source of material

The title compound was prepared according to a method reported elsewhere [1].

Experimental details

All H atoms were located in a difference Fourier synthesis.

Discussion

The unit cell contains eight molecules of the title compound. The *N*-heterocyclic carbene (*NHC*) complex exhibits a *trans* configuration of the two chloride ligands in a slightly distorted square-planar geometry around the palladium center (Cl–Pd–Cl: 174.40(2)°; C1–Pd–N3: 179.4(1)°). The carbene ring plane of the *NHC* ligand is situated almost perpendicularly to the Pd,Cl,Cl,C,N coordination plane (dihedral angle 87.4°). In contrast, the pyridine ring makes an angle of 42.4° with the metal plane, this obviously minimising its steric interactions with both the two chlorido ligands and the two benzyl groups. The most striking feature of this structure concerns the orientation of the fluorenyl planes, which are both folded back towards the C6 ring of the benzimidazolyl unit. Consequently, the benzylic methylene groups are oriented towards the d_z^2 orbital, with their hydrogen atoms coming all close to the metal centre. The corresponding $C_6H_5CH\cdots Pd$ separations (2.416-2.664 Å) lie in a range which is characteristic of anagostic M···H–C interactions [2]. In keeping with such interactions, which are essentially electrostatic in nature, the ^{1}H NMR signal of the CH_2 groups appears at much lower field than that seen for the same protons in the imidazolium precursor used for the preparation of the complex ($\Delta\delta$ = 1.75 ppm). The Pd–Cl (2.322(1) and 2.334(1) Å), Pd–C (1.954(2) Å), and Pd–N (2.093(2) Å) bond lengths are comparable to those found in related *trans*-[$PdCl_2$(*NHC*)(pyridine)] complexes [1, 3].

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(3)	16*f*	0.0771	0.4964	0.5040	0.030
H(4)	16*f*	0.0400	0.4936	0.3317	0.031
H(5)	16*f*	–0.0066	0.4595	0.3399	0.031
H(6)	16*f*	–0.0178	0.4263	0.5186	0.028
H(8)	16*f*	0.0365	0.4094	1.2374	0.041
H(9)	16*f*	0.0512	0.3915	1.4402	0.053
H(10)	16*f*	0.1065	0.3778	1.4846	0.059
H(11)	16*f*	0.1461	0.3813	1.3175	0.059
H(12)	16*f*	0.1292	0.3997	1.1174	0.047
H(15)	16*f*	0.1356	0.4278	0.6432	0.034
H(16)	16*f*	0.1797	0.4351	0.4963	0.043
H(17)	16*f*	0.1944	0.4885	0.4294	0.047
H(18)	16*f*	0.1660	0.5353	0.5034	0.040
H(21)	16*f*	0.1233	0.5804	0.6169	0.036
H(22)	16*f*	0.0798	0.6076	0.7236	0.043
H(23)	16*f*	0.0416	0.5777	0.8460	0.039
H(24)	16*f*	0.0473	0.5196	0.8713	0.031
H(26A)	16*f*	0.0980	0.4782	0.9631	0.029
H(26B)	16*f*	0.1187	0.4480	0.9013	0.029
H(28)	16*f*	0.1768	0.4522	0.8755	0.036
H(29)	16*f*	0.2272	0.4805	0.8979	0.043
H(30)	16*f*	0.2273	0.5371	0.9533	0.044
H(31)	16*f*	0.1769	0.5642	0.9916	0.042
H(32)	16*f*	0.1262	0.5360	0.9684	0.035
H(35)	16*f*	–0.0339	0.4557	0.8798	0.039
H(36)	16*f*	–0.0892	0.4725	0.8342	0.049
H(37)	16*f*	–0.1222	0.4418	0.6956	0.054

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Table 2. continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(38)	16*f*	–0.1018	0.3938	0.5963	0.046
H(41)	16*f*	–0.0641	0.3429	0.4971	0.048
H(42)	16*f*	–0.0217	0.3069	0.4314	0.057
H(43)	16*f*	0.0321	0.3116	0.5136	0.051
H(44)	16*f*	0.0443	0.3526	0.6677	0.036
H(46A)	16*f*	0.0289	0.3768	0.9103	0.030
H(46B)	16*f*	0.0023	0.4019	0.9708	0.030
H(48)	16*f*	–0.0566	0.3805	1.0086	0.044
H(49)	16*f*	–0.0853	0.3347	1.0911	0.057
H(50)	16*f*	–0.0646	0.2812	1.0661	0.057
H(51)	16*f*	–0.0143	0.2734	0.9617	0.066
H(52)	16*f*	0.0159	0.3190	0.8860	0.051

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pd(1)	16*f*	0.065917(4)	0.421353(4)	0.97556(2)	0.01938(9)	0.02056(9)	0.01670(8)	0.00058(6)	–0.00131(6)	0.00126(6)
Cl(1)	16*f*	0.03182(1)	0.46304(1)	1.05839(5)	0.0323(3)	0.0270(3)	0.0216(3)	0.0061(2)	0.0032(2)	–0.0009(2)
Cl(2)	16*f*	0.09778(1)	0.38050(1)	0.87290(5)	0.0233(3)	0.0265(3)	0.0352(3)	0.0054(2)	–0.0005(2)	–0.0035(2)
N(1)	16*f*	0.06850(4)	0.45815(4)	0.7307(2)	0.0203(8)	0.0195(8)	0.0169(8)	–0.0015(7)	–0.0003(7)	0.0003(7)
N(2)	16*f*	0.02500(4)	0.42502(4)	0.7372(2)	0.0178(8)	0.0212(8)	0.0177(9)	–0.0011(7)	0.0008(7)	0.0008(7)
N(3)	16*f*	0.08136(4)	0.40619(4)	1.1584(2)	0.032(1)	0.0250(9)	0.022(1)	–0.0006(8)	–0.0042(8)	0.0023(8)
C(1)	16*f*	0.05200(5)	0.43565(5)	0.8045(2)	0.020(1)	0.020(1)	0.019(1)	0.0046(8)	0.0021(8)	–0.0023(8)
C(2)	16*f*	0.05152(5)	0.46230(5)	0.6132(2)	0.021(1)	0.022(1)	0.019(1)	0.0020(8)	–0.0002(8)	–0.0022(8)
C(3)	16*f*	0.05816(5)	0.48222(5)	0.5069(2)	0.027(1)	0.025(1)	0.023(1)	–0.0047(9)	0.0014(9)	0.0018(9)
C(4)	16*f*	0.03607(5)	0.48048(5)	0.4061(2)	0.033(1)	0.026(1)	0.019(1)	0.0010(9)	0.0006(9)	0.0039(9)
C(5)	16*f*	0.00821(5)	0.45998(5)	0.4110(2)	0.028(1)	0.031(1)	0.019(1)	0.0049(9)	–0.0040(9)	0.0007(9)
C(6)	16*f*	0.00142(5)	0.44023(5)	0.5159(2)	0.023(1)	0.025(1)	0.021(1)	0.0000(9)	–0.0004(9)	–0.0013(9)
C(7)	16*f*	0.02385(5)	0.44151(5)	0.6176(2)	0.022(1)	0.020(1)	0.017(1)	0.0022(8)	0.0015(8)	–0.0004(8)
C(8)	16*f*	0.05913(6)	0.40367(6)	1.2539(2)	0.043(1)	0.033(1)	0.028(1)	0.013(1)	0.003(1)	0.005(1)
C(9)	16*f*	0.06774(7)	0.39310(6)	1.3750(2)	0.067(2)	0.043(2)	0.023(1)	0.022(1)	0.007(1)	0.007(1)
C(10)	16*f*	0.10006(7)	0.38491(7)	1.4011(2)	0.071(2)	0.050(2)	0.025(1)	0.006(1)	–0.017(1)	0.005(1)
C(11)	16*f*	0.12346(7)	0.38713(7)	1.3029(3)	0.043(2)	0.058(2)	0.048(2)	–0.002(1)	–0.022(1)	0.012(1)
C(12)	16*f*	0.11312(6)	0.39795(6)	1.1841(2)	0.029(1)	0.052(2)	0.037(2)	–0.005(1)	–0.010(1)	0.012(1)
C(13)	16*f*	0.09793(5)	0.47962(5)	0.7613(2)	0.022(1)	0.022(1)	0.019(1)	–0.0051(8)	–0.0017(8)	–0.0010(8)
C(14)	16*f*	0.12492(5)	0.47739(5)	0.6595(2)	0.022(1)	0.028(1)	0.018(1)	–0.0034(9)	–0.0025(8)	–0.0026(9)
C(15)	16*f*	0.14172(5)	0.44952(6)	0.6151(2)	0.029(1)	0.031(1)	0.026(1)	–0.001(1)	–0.002(1)	–0.004(1)
C(16)	16*f*	0.16788(6)	0.45390(6)	0.5281(2)	0.032(1)	0.044(1)	0.033(1)	0.002(1)	0.005(1)	–0.012(1)
C(17)	16*f*	0.17650(6)	0.48576(7)	0.4883(2)	0.031(1)	0.057(2)	0.029(1)	–0.010(1)	0.010(1)	–0.010(1)
C(18)	16*f*	0.15976(6)	0.51360(6)	0.5319(2)	0.034(1)	0.039(1)	0.027(1)	–0.013(1)	0.002(1)	–0.000(1)
C(19)	16*f*	0.13372(5)	0.50949(5)	0.6182(2)	0.026(1)	0.030(1)	0.018(1)	–0.0076(9)	–0.0012(9)	–0.0018(9)
C(20)	16*f*	0.11061(5)	0.53371(5)	0.6778(2)	0.028(1)	0.026(1)	0.019(1)	–0.0058(9)	–0.0021(9)	–0.0003(9)
C(21)	16*f*	0.10781(6)	0.56807(5)	0.6670(2)	0.038(1)	0.026(1)	0.027(1)	–0.009(1)	0.000(1)	0.002(1)
C(22)	16*f*	0.08207(6)	0.58403(6)	0.7306(2)	0.046(2)	0.023(1)	0.039(1)	–0.003(1)	–0.005(1)	–0.001(1)
C(23)	16*f*	0.05940(6)	0.56622(5)	0.8047(2)	0.032(1)	0.030(1)	0.036(1)	0.004(1)	–0.001(1)	–0.007(1)
C(24)	16*f*	0.06245(5)	0.53181(5)	0.8192(2)	0.027(1)	0.028(1)	0.024(1)	–0.0011(9)	–0.0004(9)	–0.0025(9)
C(25)	16*f*	0.08820(5)	0.51611(5)	0.7555(2)	0.026(1)	0.022(1)	0.019(1)	–0.0032(9)	–0.0035(9)	–0.0018(8)
C(26)	16*f*	0.11409(5)	0.47220(5)	0.8946(2)	0.026(1)	0.025(1)	0.020(1)	–0.0039(9)	–0.0006(9)	0.0015(9)
C(27)	16*f*	0.14628(5)	0.49131(5)	0.9156(2)	0.027(1)	0.029(1)	0.016(1)	–0.0060(9)	–0.0032(9)	0.0034(9)
C(28)	16*f*	0.17666(5)	0.47522(6)	0.8977(2)	0.033(1)	0.030(1)	0.025(1)	–0.001(1)	–0.006(1)	–0.002(1)
C(29)	16*f*	0.20672(6)	0.49191(6)	0.9115(2)	0.024(1)	0.050(2)	0.033(1)	–0.003(1)	–0.003(1)	–0.004(1)
C(30)	16*f*	0.20676(6)	0.52530(6)	0.9451(2)	0.030(1)	0.050(2)	0.030(1)	–0.014(1)	–0.004(1)	–0.003(1)
C(31)	16*f*	0.17684(6)	0.54141(6)	0.9668(2)	0.041(1)	0.036(1)	0.027(1)	–0.014(1)	–0.004(1)	–0.007(1)
C(32)	16*f*	0.14662(6)	0.52465(5)	0.9527(2)	0.031(1)	0.033(1)	0.022(1)	–0.004(1)	–0.0010(9)	–0.0042(9)
C(33)	16*f*	–0.00084(5)	0.39945(5)	0.7718(2)	0.018(1)	0.024(1)	0.022(1)	–0.0040(8)	0.0004(8)	0.0027(9)
C(34)	16*f*	–0.03567(5)	0.41466(5)	0.7630(2)	0.021(1)	0.031(1)	0.025(1)	0.0006(9)	0.0038(9)	0.0101(9)
C(35)	16*f*	–0.04754(6)	0.44321(6)	0.8225(2)	0.031(1)	0.035(1)	0.032(1)	0.004(1)	0.009(1)	0.008(1)
C(36)	16*f*	–0.08036(6)	0.45299(6)	0.7952(3)	0.036(1)	0.042(1)	0.044(2)	0.013(1)	0.018(1)	0.018(1)
C(37)	16*f*	–0.09994(6)	0.43464(7)	0.7124(3)	0.022(1)	0.062(2)	0.052(2)	0.006(1)	0.004(1)	0.029(2)
C(38)	16*f*	–0.08805(5)	0.40618(7)	0.6532(2)	0.021(1)	0.054(2)	0.039(2)	–0.007(1)	–0.002(1)	0.022(1)
C(39)	16*f*	–0.05547(5)	0.39599(6)	0.6784(2)	0.022(1)	0.036(1)	0.027(1)	–0.008(1)	0.0003(9)	0.015(1)
C(40)	16*f*	–0.03505(5)	0.36919(5)	0.6230(2)	0.027(1)	0.031(1)	0.027(1)	–0.0102(9)	–0.0057(9)	0.009(1)
C(41)	16*f*	–0.04224(6)	0.34491(6)	0.5319(2)	0.043(1)	0.044(2)	0.034(1)	–0.020(1)	–0.012(1)	0.004(1)
C(42)	16*f*	–0.01700(7)	0.32374(6)	0.4931(3)	0.064(2)	0.037(1)	0.041(2)	–0.016(1)	–0.008(1)	–0.012(1)
C(43)	16*f*	0.01515(7)	0.32642(6)	0.5422(2)	0.055(2)	0.030(1)	0.042(2)	–0.002(1)	0.003(1)	–0.007(1)
C(44)	16*f*	0.02243(6)	0.35073(5)	0.6329(2)	0.033(1)	0.027(1)	0.031(1)	–0.003(1)	–0.000(1)	0.000(1)
C(45)	16*f*	–0.00251(5)	0.37197(5)	0.6716(2)	0.026(1)	0.024(1)	0.020(1)	–0.0056(9)	–0.0004(9)	0.0047(9)
C(46)	16*f*	0.00533(5)	0.38411(5)	0.9058(2)	0.025(1)	0.030(1)	0.021(1)	–0.0050(9)	–0.0014(9)	0.0024(9)
C(47)	16*f*	–0.01660(5)	0.35487(5)	0.9426(2)	0.026(1)	0.030(1)	0.021(1)	–0.0045(9)	–0.0058(9)	0.0057(9)
C(48)	16*f*	–0.04723(6)	0.35883(6)	1.0007(2)	0.037(1)	0.034(1)	0.040(2)	–0.007(1)	0.005(1)	0.000(1)
C(49)	16*f*	–0.06464(6)	0.33134(7)	1.0482(3)	0.038(1)	0.054(2)	0.052(2)	–0.016(1)	0.005(1)	0.006(1)
C(50)	16*f*	–0.05247(7)	0.29981(7)	1.0340(3)	0.048(2)	0.041(2)	0.052(2)	–0.017(1)	–0.013(1)	0.021(1)
C(51)	16*f*	–0.02282(7)	0.29537(7)	0.9733(3)	0.063(2)	0.028(1)	0.075(2)	0.002(1)	–0.005(2)	0.017(1)
C(52)	16*f*	–0.00488(6)	0.32260(6)	0.9280(3)	0.038(1)	0.037(1)	0.051(2)	0.006(1)	0.004(1)	0.012(1)

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