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Crystal structure of dichlorobis[*N*-(2,6-diisopropylphenyl)-benzimidimidazolide]palladium(II), $PdCl_2(C_{22}H_{25}N_3)_2$

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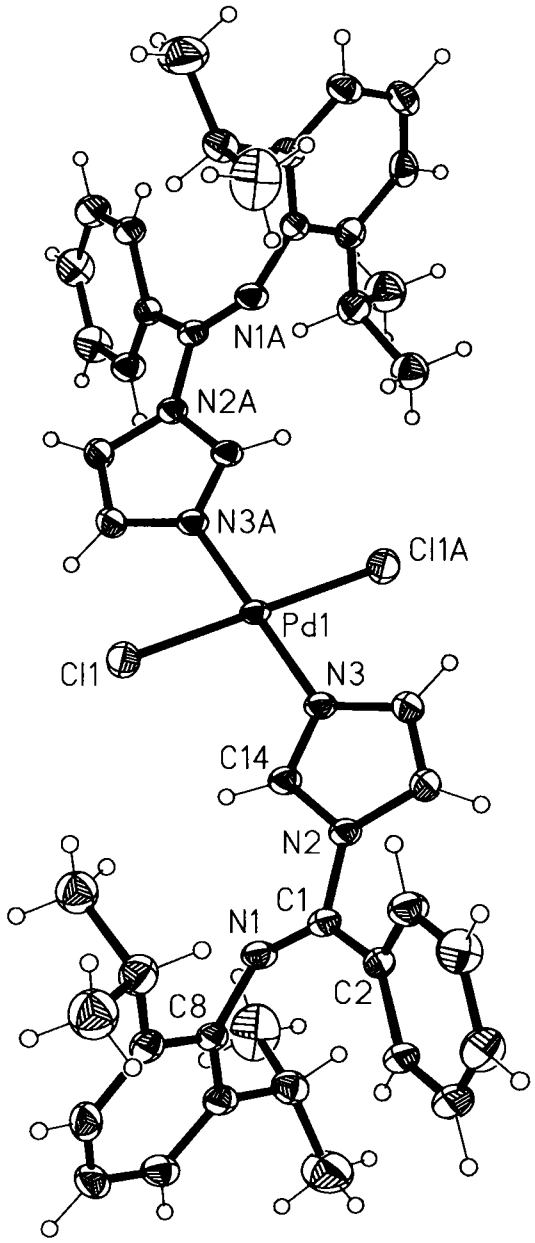

Abstract

$C_{44}H_{50}Cl_2N_6Pd$, monoclinic, $P12_1/c1$ (no. 14), a = 8.9983(2) Å, b = 12.9387(3) Å, c = 18.6706(4) Å, β = 101.365(2)°, V = 2131.1 Å^3, Z = 2, $R_{gt}(F)$ = 0.027, $wR_{ref}(F^2)$ = 0.072, T = 233 K.

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Source of material

The Pd complex was obtained from the corresponding imidazole and $PdCl_2$ in an attempt to form a *N*-heterocyclic carbene complex [1] following Raubenheimer's methodology [2].

Experimental details

All hydrogen atoms were calculated and refined using a riding model. The methyl carbon atoms at one isopropyl group have an 1:1 position disorder (C92, C93 : C92A, C93A). For hydrogen calculation the methine carbon atom were divided in C91 and C91A with equal coordinates and displacement factors, but each with an occupancy of 0.5.

Discussion

The asymmetric unit is occupied with one half molecule, whereas the Pd(II) cation lies in a symmetry center with a square planar coordination and Pd—N and Pd—Cl distances of 2.024(2) Å and 2.306(1) Å, respectively. The imidazolide rings are not coplanar, represented by a torsion angle Cl1–Pd1–N3–C14 of 24.1(2)°. The environment around the imino function with a double bond C1=N1 of 1.261(3) Å can be described by dihedral angles from the main plane (C8, N1, C1, C2, N2) to the diisopropylphenyl-, phenyl- and imidazole ring with 76.1(1)°, 40.6(1)° and 23.6(1)°, respectively. A comparison with the structure of the free ligand [3] with a double bond of 1.272(3) Å and dihedral angles of 72.9(1)°, 45.7(1)° and 23.1(1)° shows a small influence of the Pd-coordination by reducing the length of the double bond. Interestingly, the double bond reduces to 1.248(2) Å by exchanging the imidazole ring of the free ligand through an electron-withdrawing chlorine atom [1]. The dihedral angle of the diisopropylphenyl- and phenyl ring changes to 80.7(1)° and 3.0(1)°. By coordination of N1 to the Pd(II) cation, forming a bidentate nitrogen *cis*-Pd(II) complex [4], the double bond can be significantly increased to 1.303(3) Å, shown by a similar ligand with small variations (C14 bounds to C1 and one nitrogen atom of the imidazole ring is methylated.).

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.1 × 0.3 × 0.3 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	5.98 cm^{-1}
Diffractometer, scan mode:	Nonius KappaCCD, φ/ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	13239, 3764
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3293
$N(param)_{refined}$:	240
Programs:	SHELXS-97 [5], SHELXL-97 [6], SHELXTL [7]

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

Atom	Site	Occ.	x	y	z	U_{iso}
H(3)	4e		−0.0654	0.3246	−0.2991	0.043
H(4)	4e		−0.3080	0.3922	−0.3140	0.057
H(5)	4e		−0.4309	0.3990	−0.2162	0.061
H(6)	4e		−0.3120	0.3363	−0.1027	0.058
H(7)	4e		−0.0719	0.2641	−0.0873	0.046
H(10)	4e		0.3059	0.6176	−0.2306	0.047
H(11)	4e		0.3558	0.5961	−0.3461	0.051
H(12)	4e		0.3097	0.4388	−0.4050	0.047
H(14)	4e		0.3816	0.1833	−0.1042	0.036
H(15)	4e		0.1320	−0.0444	−0.0509	0.041
H(16)	4e		−0.0398	0.0726	−0.1320	0.043
H(131)	4e		0.1549	0.2165	−0.3332	0.047
H(13A)	4e		0.3588	0.1249	−0.3648	0.119
H(13B)	4e		0.4088	0.1833	−0.2893	0.119
H(13C)	4e		0.4528	0.2290	−0.3611	0.119
H(13D)	4e		0.1486	0.1885	−0.4576	0.112
H(13E)	4e		0.2271	0.2970	−0.4631	0.112
H(13F)	4e		0.0621	0.2918	−0.4455	0.112

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
H(91)	4e	0.5	0.1437	0.4333	−0.1280	0.052
C(92)	4e	0.5	0.3760(6)	0.4536(5)	−0.0817(3)	0.049(1)
H(92A)	4e	0.5	0.3623	0.4616	−0.0317	0.073
H(92B)	4e	0.5	0.4563	0.4992	−0.0902	0.073
H(92C)	4e	0.5	0.4029	0.3826	−0.0897	0.073
C(93)	4e	0.5	0.1763(8)	0.5917(5)	−0.1187(3)	0.065(2)
H(93A)	4e	0.5	0.1626	0.5946	−0.0685	0.097
H(93B)	4e	0.5	0.0820	0.6095	−0.1513	0.097
H(93C)	4e	0.5	0.2546	0.6402	−0.1254	0.097
H(91A)	4e	0.5	0.1932	0.4135	−0.1204	0.052
C(93A)	4e	0.5	0.0854(9)	0.5558(6)	−0.1360(4)	0.077(2)
H(93D)	4e	0.5	−0.0002	0.5327	−0.1726	0.115
H(93E)	4e	0.5	0.1125	0.6257	−0.1474	0.115
H(93F)	4e	0.5	0.0583	0.5548	−0.0883	0.115
C(92A)	4e	0.5	0.3574(9)	0.5133(6)	−0.0818(4)	0.075(2)
H(92D)	4e	0.5	0.3303	0.5194	−0.0342	0.112
H(92E)	4e	0.5	0.3942	0.5793	−0.0957	0.112
H(92F)	4e	0.5	0.4362	0.4616	−0.0796	0.112

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

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pd(1)	2b		½	0	0	0.0229(1)	0.0263(1)	0.0221(1)	0.00436(8)	0.00255(9)	0.00607(8)
Cl(1)	4e		0.60557(7)	0.16303(4)	0.01062(3)	0.0395(3)	0.0302(3)	0.0538(3)	−0.0013(2)	−0.0064(3)	0.0081(2)
N(1)	4e		0.2244(2)	0.2808(1)	−0.20789(9)	0.028(1)	0.0358(9)	0.0295(9)	0.0056(8)	0.0042(7)	0.0091(8)
N(2)	4e		0.1537(2)	0.1605(1)	−0.13425(9)	0.0227(9)	0.0325(9)	0.0298(9)	0.0039(7)	0.0015(7)	0.0091(7)
N(3)	4e		0.3065(2)	0.0557(1)	−0.06199(9)	0.0263(9)	0.0306(9)	0.0268(8)	0.0038(7)	0.0031(7)	0.0070(7)
C(1)	4e		0.1140(2)	0.2476(2)	−0.1822(1)	0.027(1)	0.030(1)	0.026(1)	0.0032(8)	0.0009(8)	0.0043(8)
C(2)	4e		−0.0429(2)	0.2869(2)	−0.1915(1)	0.022(1)	0.028(1)	0.034(1)	0.0017(8)	0.0007(8)	0.0060(9)
C(3)	4e		−0.1152(2)	0.3261(2)	−0.2594(1)	0.028(1)	0.041(1)	0.036(1)	0.0019(9)	−0.0012(9)	0.012(1)
C(4)	4e		−0.2596(3)	0.3669(2)	−0.2681(1)	0.032(1)	0.052(1)	0.052(1)	0.007(1)	−0.004(1)	0.019(1)
C(5)	4e		−0.3330(3)	0.3708(2)	−0.2100(2)	0.026(1)	0.058(2)	0.069(2)	0.014(1)	0.007(1)	0.014(1)
C(6)	4e		−0.2622(3)	0.3330(2)	−0.1424(1)	0.037(1)	0.057(2)	0.055(2)	0.009(1)	0.018(1)	0.006(1)
C(7)	4e		−0.1186(2)	0.2905(2)	−0.1331(1)	0.033(1)	0.044(1)	0.037(1)	0.006(1)	0.005(1)	0.008(1)
C(8)	4e		0.2327(2)	0.3723(2)	−0.2485(1)	0.017(1)	0.033(1)	0.035(1)	0.0040(8)	0.0017(8)	0.0111(9)
C(9)	4e		0.2484(2)	0.4690(2)	−0.2136(1)	0.022(1)	0.040(1)	0.039(1)	0.0037(9)	0.0018(9)	0.006(1)
C(10)	4e		0.2937(2)	0.5520(2)	−0.2526(1)	0.026(1)	0.035(1)	0.056(2)	−0.0012(9)	0.002(1)	0.004(1)
C(11)	4e		0.3206(3)	0.5400(2)	−0.3221(1)	0.028(1)	0.043(1)	0.056(2)	−0.002(1)	0.008(1)	0.019(1)
C(12)	4e		0.2960(2)	0.4454(2)	−0.3565(1)	0.030(1)	0.049(1)	0.039(1)	0.004(1)	0.0098(9)	0.015(1)
C(13)	4e		0.2515(2)	0.3596(2)	−0.3211(1)	0.023(1)	0.041(1)	0.035(1)	0.0051(9)	0.0060(9)	0.010(1)
C(14)	4e		0.2982(2)	0.1412(2)	−0.1005(1)	0.023(1)	0.037(1)	0.030(1)	0.0037(9)	0.0042(8)	0.0088(9)
C(15)	4e		0.1605(3)	0.0167(2)	−0.0718(1)	0.029(1)	0.034(1)	0.039(1)	−0.0023(9)	0.003(1)	0.0098(9)
C(16)	4e		0.0655(2)	0.0807(2)	−0.1163(1)	0.025(1)	0.037(1)	0.043(1)	−0.0017(9)	−0.0007(9)	0.011(1)
C(131)	4e		0.2282(3)	0.2539(2)	−0.3568(1)	0.040(1)	0.042(1)	0.039(1)	0.001(1)	0.013(1)	0.005(1)
C(132)	4e		0.3756(4)	0.1921(3)	−0.3416(2)	0.062(2)	0.061(2)	0.114(3)	0.019(2)	0.018(2)	−0.016(2)
C(133)	4e		0.1604(4)	0.2582(3)	−0.4381(2)	0.116(3)	0.064(2)	0.042(2)	−0.012(2)	0.010(2)	−0.003(1)
C(91)	4e	0.5	0.2236(3)	0.4822(2)	−0.1359(1)	0.043(1)	0.047(1)	0.040(1)	−0.003(1)	0.006(1)	−0.003(1)
C(91A)	4e	0.5	0.2236(3)	0.4822(2)	−0.1359(1)	0.043(1)	0.047(1)	0.040(1)	−0.003(1)	0.006(1)	−0.003(1)

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