Crystal structure of dichloro((*S*)-2-(anilinomethyl)-pyrrolidine)-palladium(II), $Pd(Cl)_2(C_{11}H_{16}N_2)$

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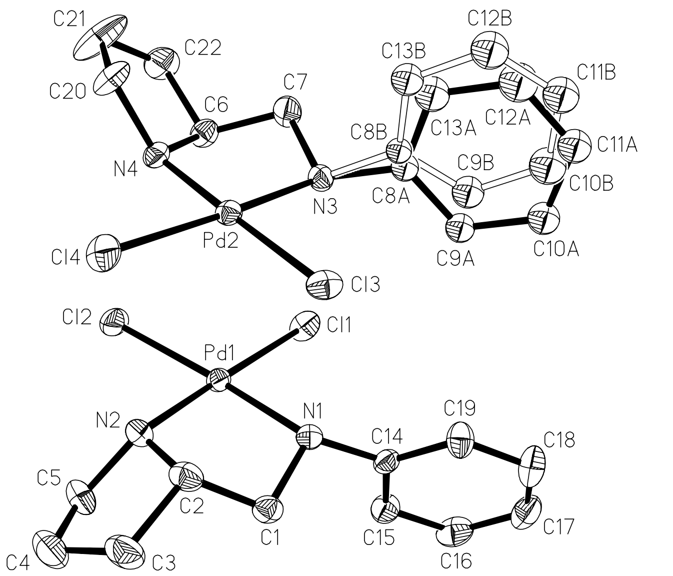

Abstract

$C_{11}H_{16}Cl_2N_2Pd$, orthorhombic, $P2_12_12_1$ (no. 19), a = 11.246(2) Å, b = 12.222(2) Å, c = 19.637(4) Å, V = 2699.1 Å^3, Z = 8, $R_{gt}(F)$ = 0.027, $wR_{ref}(F^2)$ = 0.060, T = 200 K.

Source of material

The title compound was prepared by reaction of Li_2PdCl_4 and (*S*)-2-(anilinomethyl)-pyrrolidine (molar ratio 1:1) in distilled water at 298 K. The resulting yellow solid, after concentration to dryness, was treated with a mixture of CH_2Cl_2/CH_3OH. After filtration from the resulting solution, crystals of the complex were obtained.

Experimental details

All non-hydrogen atoms, except the disordered C atoms which were refined only isotropically, were refined anisotropically. The H atoms bound to C atoms were included at calculated positions and refined by using the riding model. The H atoms attached to N atoms were found from Fourier difference maps and refined freely.

Discussion

Chiral chelating bidentate nitrogen ligands have found widespread application in asymmetric homogeneous catalysis by transition metal complexes [1,2]. Their palladium complexes are used as efficient catalyst in a number of reactions such as green oxidations of alcohols in an aqueous biphasic system [3] and enantio-selective allylic alkylation [4]. As part of our research on transition metal complexes and their application in catalytic valorisation of natural terpenes [5-9], and before employing any catalytic procedures, we try to obtain a palladium complex of dichloro((*S*)-2-(anilinomethyl)-pyrrolidine.

The environment of palladium atom is made up of (*S*)-2-(anilinomethyl)-pyrrolidine linked in bidentate manner through it two nitrogen atoms, defining a five-membered chelate ring and two chlorine atoms. The phenyl ring in one of the both molecules of the asymmetric unit is disordered. The main difference of the two molecules is in the angles between the coordination planes (Pd1, Cl1, Cl2, N1, N2 and Pd2, Cl3, Cl4, N3, N4, respectively) and the planes defined by the phenyl rings being 73.0(2)° for the first molecule and 82.8(2)°/86.7(2)° for the second, respectively.

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Table 1. Data collection and handling.

Crystal:	orange prism, size 0.4 × 0.5 × 0.5 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	17.46 cm^{-1}
Diffractometer, scan mode:	Stoe IPDS I, φ
$2\theta_{max}$:	52.3°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	17588, 5296
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4797
$N(param)_{refined}$:	276
Programs:	SHELXS-97 [10], SHELXL-97 [11]

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

Atom	Site	Occ.	x	y	z	U_{iso}
H(1A)	4*a*		0.2847	0.1045	1.0717	0.044
H(1B)	4*a*		0.2279	−0.0157	1.0776	0.044
H(2)	4*a*		0.1070	0.1743	1.0255	0.051
H(3A)	4*a*		0.1093	0.0953	1.1554	0.071
H(3B)	4*a*		0.0189	0.1866	1.1270	0.071
H(4A)	4*a*		−0.0490	−0.0108	1.1647	0.080
H(4B)	4*a*		−0.1270	0.0669	1.1161	0.080
H(5A)	4*a*		−0.1062	−0.0772	1.0465	0.072
H(5B)	4*a*		0.0227	−0.1084	1.0768	0.072
H(6)	4*a*		0.0270	−0.0628	0.7528	0.047
H(7A)	4*a*		0.1684	0.0181	0.6806	0.053
H(7B)	4*a*		0.1001	0.1335	0.6825	0.053
C(8A)	4*a*	0.56(1)	0.3027(4)	0.1454(5)	0.7551(3)	0.035(2)
C(9A)	4*a*	0.56	0.4063(5)	0.1040(4)	0.7838(3)	0.040(2)
H(9A)	4*a*	0.56	0.4028	0.0422	0.8131	0.048
C(10A)	4*a*	0.56	0.5150(4)	0.1532(5)	0.7698(3)	0.048(2)
H(10A)	4*a*	0.56	0.5859	0.1249	0.7895	0.058
C(11A)	4*a*	0.56	0.5201(4)	0.2437(5)	0.7271(3)	0.050(2)
H(11A)	4*a*	0.56	0.5945	0.2773	0.7175	0.060
C(12A)	4*a*	0.56	0.4165(6)	0.2851(5)	0.6983(3)	0.067(3)
H(12A)	4*a*	0.56	0.4200	0.3470	0.6691	0.080
C(13A)	4*a*	0.56	0.3078(4)	0.2359(6)	0.7124(3)	0.054(2)
H(13A)	4*a*	0.56	0.2370	0.2642	0.6927	0.065

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
C(8B)	4*a*	0.44	0.2856(5)	0.1763(6)	0.7511(3)	0.029(2)
C(9B)	4*a*	0.44	0.4014(6)	0.1396(5)	0.7605(4)	0.048(3)
H(9B)	4*a*	0.44	0.4157	0.0718	0.7826	0.057
C(10B)	4*a*	0.44	0.4964(5)	0.2023(6)	0.7375(4)	0.061(4)
H(10B)	4*a*	0.44	0.5756	0.1772	0.7439	0.074
C(11B)	4*a*	0.44	0.4756(6)	0.3016(6)	0.7051(4)	0.056(3)
H(11B)	4*a*	0.44	0.5405	0.3444	0.6894	0.068
C(12B)	4*a*	0.44	0.3597(7)	0.3382(5)	0.6957(3)	0.059(3)
H(12B)	4*a*	0.44	0.3455	0.4061	0.6736	0.071
C(13B)	4*a*	0.44	0.2647(5)	0.2756(6)	0.7187(3)	0.045(3)
H(13B)	4*a*	0.44	0.1855	0.3006	0.7123	0.054
H(15)	4*a*		0.3552	–0.1643	1.0125	0.051
H(16)	4*a*		0.5527	–0.2201	1.0114	0.065

Table 2. Continued.

Atom	Site	Occ.	x	y	z	U_{iso}
H(17)	4*a*		0.7010	–0.1035	0.9757	0.068
H(18)	4*a*		0.6539	0.0717	0.9404	0.070
H(19)	4*a*		0.4563	0.1310	0.9412	0.053
H(20A)	4*a*		–0.2063	0.1696	0.7763	0.071
H(20B)	4*a*		–0.1016	0.2174	0.7289	0.071
H(21A)	4*a*		–0.1951	0.1230	0.6501	0.122
H(21B)	4*a*		–0.2502	0.0389	0.7042	0.122
H(22A)	4*a*		–0.0457	0.0168	0.6303	0.070
H(22B)	4*a*		–0.1152	–0.0758	0.6729	0.070
HN(1)	4*a*		0.257(5)	0.093(4)	0.959(2)	0.06(2)
HN(2)	4*a*		–0.016(4)	0.067(4)	0.990(2)	0.04(1)
HN(3)	4*a*		0.201(4)	0.033(4)	0.792(2)	0.03(1)
HN(4)	4*a*		–0.087(4)	0.035(3)	0.816(2)	0.03(1)

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}
C(1)	4*a*	0.2282(4)	0.0526(4)	1.0504(2)	0.042(2)	0.037(2)	0.031(2)	–0.004(2)	–0.003(2)	–0.004(2)
C(2)	4*a*	0.1055(4)	0.1015(4)	1.0488(2)	0.052(3)	0.043(2)	0.033(2)	0.007(2)	0.004(2)	–0.000(2)
C(3)	4*a*	0.0499(5)	0.1118(5)	1.1196(2)	0.070(4)	0.071(3)	0.036(2)	0.013(3)	0.012(2)	–0.004(2)
C(4)	4*a*	–0.0494(5)	0.0296(5)	1.1210(2)	0.067(4)	0.078(4)	0.057(3)	0.019(3)	0.026(3)	0.022(3)
C(5)	4*a*	–0.0299(5)	–0.0472(5)	1.0633(2)	0.049(3)	0.082(3)	0.049(2)	–0.011(3)	0.017(2)	0.011(3)
C(6)	4*a*	0.0081(4)	0.0104(4)	0.7328(2)	0.039(2)	0.041(2)	0.039(2)	0.002(2)	–0.005(2)	–0.006(2)
C(7)	4*a*	0.1206(4)	0.0678(4)	0.7095(2)	0.041(2)	0.059(3)	0.033(2)	–0.002(2)	0.003(2)	–0.012(2)
C(14)	4*a*	0.3873(4)	–0.0105(3)	0.9758(2)	0.031(2)	0.035(2)	0.031(2)	–0.003(2)	–0.007(2)	–0.000(1)
C(15)	4*a*	0.4157(4)	–0.1158(3)	0.9975(2)	0.042(3)	0.035(2)	0.050(2)	–0.001(2)	–0.005(2)	0.008(2)
C(16)	4*a*	0.5328(5)	–0.1482(4)	0.9969(2)	0.060(3)	0.046(3)	0.056(3)	0.023(3)	–0.011(2)	0.005(2)
C(17)	4*a*	0.6206(5)	–0.0796(4)	0.9759(2)	0.037(2)	0.072(3)	0.060(3)	0.007(3)	–0.010(2)	–0.011(2)
C(18)	4*a*	0.5927(4)	0.0236(4)	0.9551(3)	0.034(3)	0.060(3)	0.080(3)	–0.008(2)	0.001(2)	–0.009(2)
C(19)	4*a*	0.4753(4)	0.0588(4)	0.9553(2)	0.038(2)	0.034(2)	0.060(3)	–0.005(2)	0.003(2)	–0.002(2)
C(20)	4*a*	–0.1384(5)	0.1497(4)	0.7467(3)	0.046(3)	0.066(3)	0.065(3)	0.016(3)	–0.026(2)	–0.003(2)
C(21)	4*a*	–0.1770(7)	0.0784(6)	0.6908(3)	0.100(5)	0.089(5)	0.116(5)	0.039(4)	–0.080(5)	–0.044(4)
C(22)	4*a*	–0.0828(5)	–0.0006(4)	0.6747(2)	0.062(3)	0.067(3)	0.046(2)	–0.017(3)	–0.016(2)	–0.009(2)
Pd(1)	4*a*	0.12168(3)	–0.05039(2)	0.93516(1)	0.0282(1)	0.0300(1)	0.0304(1)	–0.0017(1)	–0.0030(1)	0.0036(1)
Cl(1)	4*a*	0.2347(1)	–0.12799(8)	0.85067(5)	0.0435(6)	0.0398(5)	0.0458(5)	0.0108(5)	–0.0064(4)	–0.0141(4)
Cl(2)	4*a*	–0.0530(1)	–0.1239(1)	0.89283(5)	0.0391(6)	0.0596(6)	0.0499(6)	–0.0171(5)	–0.0124(5)	0.0129(5)
N(1)	4*a*	0.2653(3)	0.0287(2)	0.9790(2)	0.032(2)	0.023(2)	0.030(2)	0.002(1)	–0.001(1)	0.002(1)
N(2)	4*a*	0.0280(4)	0.0233(3)	1.0104(2)	0.037(2)	0.060(3)	0.034(2)	0.002(2)	0.006(2)	0.010(2)
Pd(2)	4*a*	0.07171(3)	0.16569(2)	0.84010(1)	0.0310(2)	0.0273(1)	0.0296(1)	0.0065(1)	–0.0002(1)	0.0012(1)
Cl(3)	4*a*	0.2180(1)	0.25531(8)	0.90126(5)	0.0527(7)	0.0278(5)	0.0388(5)	–0.0036(5)	–0.0086(4)	0.0020(4)
Cl(4)	4*a*	–0.0783(1)	0.2186(1)	0.91289(5)	0.0489(7)	0.0616(7)	0.0512(6)	0.0289(6)	0.0067(5)	–0.0095(5)
N(3)	4*a*	0.1905(3)	0.1003(3)	0.7711(2)	0.032(2)	0.040(2)	0.030(2)	–0.002(2)	0.002(1)	–0.001(1)
N(4)	4*a*	–0.0503(3)	0.0828(3)	0.7844(2)	0.031(2)	0.041(2)	0.037(2)	0.009(2)	–0.003(1)	0.004(1)

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