Crystal structure of *trans*-dichloro-bis[(*E*)-2-(1-methoxy-imidazol-2-yl)-2-oxoacetaldehyde *O*-methyl oxime]palladium(II), $PdCl_2(C_7H_9N_3O_3)_2$

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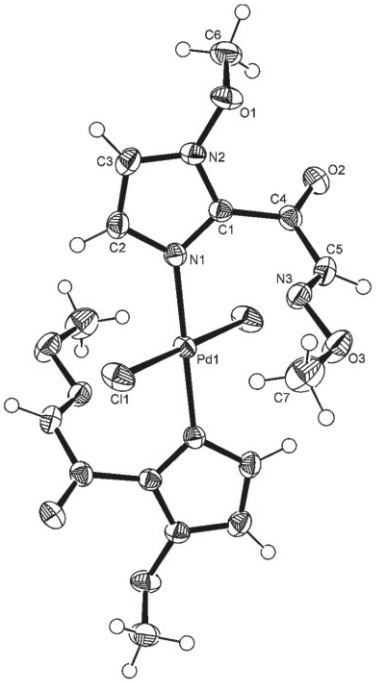

Abstract

$C_{14}H_{18}Cl_2N_6O_6Pd$, triclinic, $P\bar{1}$ (no. 2), a = 7.4741(5) Å, b = 8.3215(5) Å, c = 9.1618(6) Å, α = 73.605(3)°, β = 76.146(4)°, γ = 70.823(4)°, V = 509.5 Å^3, Z = 1, $R_{gt}(F)$ = 0.020, $wR_{ref}(F^2)$ = 0.048, T = 233 K.

Source of material

A mixture of $PdCl_2$ and (*E*)-2-(1-methoxyimidazol-2-yl)-2-oxoacetaldehyde *O*-methyl oxime [1] (2 equiv.) in MeOH was stirred for 4 h at room temperature. The product precipitated and was filtered off and washed with MeOH and Et_2O. Yellow single crystals were obtained by slow evaporation of a solution in CH_3CN (yield 71 %, m.p. 195 °C).

Discussion

The Pd atom occupies an inversion center and is coordinated in a square-planar fashion with distances of *d*(Pd—Cl) = 2.300 Å and *d*(Pd—N) = 2.016 Å. The 1,2-disubstituted imidazole coordinates with the electron pair at N1. The dihedral angle between the imidazole ring and the ligand plane is 47°. Similar *trans*-dichloro-bis(imidazole)-Pd(II) (not counting benzimidazole) complexes have been reported previously [2-13]. However, complexes involving 1,2-disubstituted imidazole ligands seem to be rare [14-17]. The O1–C6 bond of the methoxy group is twisted out of the imidazole plane by 80°, and the oxygen atom O1 is also bent out of this plane showing a distance of 0.192 Å from the imidazole ring. The side chain C4–C5–N3–O3–C7 adopts an almost planar conformation (deviations: C4, 0.012 Å; C5, 0.006 Å; N3, 0.018 Å; O3, 0.007 Å; C7, 0.006 Å), only the carbonyl oxygen atom O2 exhibits a distance of 0.417 Å from this plane. The most striking difference between the structures of the free ligand [1] and the coordinated ligand is the orientation of the side chain. In the title compound, the C4–C5 bond is rotated out of the imidazole ring plane by 50°, whereas in the free ligand it remains coplanar with the heterocyclic ring. The ligands are interconnected by weak hydrogen bonds: C3–H···O2^{i} (*d*(H···acceptor) = 2.58 Å and *d*(donor···acceptor) = 3.253 Å, ∠donor–H···acceptor = 129°) and C7–H···O3^{ii} (2.56 Å and 3.348 Å, 138°). The nitrogen atom N3 of the side chain does not participate in any interaction which is in contrast to the situation in the free ligand [1]. The imidazole rings are oriented in a parallel manner, stacked in the direction of the crystallographic *b* axis, with an interplanar distance of 8.14 Å. Symmetry codes i: 1+*x*,*y*,*z*; ii: –1–*x*,1–*y*,–*z*.

Table 1. Data collection and handling.

Crystal:	yellow prism, size 0.06 × 0.08 × 0.15 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	12.17 cm^{-1}
Diffractometer, scan mode:	Nonius KappaCCD, φ/ω
$2\theta_{max}$:	52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3118, 1970
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1906
$N(param)_{refined}$:	136
Programs:	SHELXS-97, SHELXL-97 [18]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(2)	2*i*	0.3324	–0.1479	0.2066	0.036
H(3)	2*i*	0.2850	–0.2574	0.4886	0.038
H(5)	2*i*	–0.5282	0.0753	0.2449	0.038
H(6A)	2*i*	–0.1575	–0.4499	0.6383	0.060
H(6B)	2*i*	–0.1223	–0.4290	0.7950	0.060
H(6C)	2*i*	0.0542	–0.4810	0.6650	0.060
H(7A)	2*i*	–0.3717	0.5052	0.2491	0.079
H(7B)	2*i*	–0.4308	0.5807	0.0828	0.079
H(7C)	2*i*	–0.2314	0.4389	0.1052	0.079

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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)	1*a*	0	0	0	0.0246(1)	0.0247(1)	0.0218(1)	–0.01089(9)	–0.00122(8)	–0.00515(8)
Cl(1)	2*i*	0.17900(9)	0.18608(7)	–0.02165(6)	0.0526(4)	0.0431(3)	0.0328(3)	–0.0327(3)	–0.0047(2)	–0.0062(2)
N(1)	2*i*	0.0408(2)	–0.1033(2)	0.2208(2)	0.0235(8)	0.0255(8)	0.0254(8)	–0.0095(7)	–0.0039(6)	–0.0046(6)
N(2)	2*i*	0.0007(2)	–0.2050(2)	0.4682(2)	0.0311(9)	0.0289(8)	0.0208(8)	–0.0099(7)	–0.0040(7)	–0.0045(7)
N(3)	2*i*	–0.3375(2)	0.1864(2)	0.2461(2)	0.0269(9)	0.0316(9)	0.0297(9)	–0.0027(7)	–0.0070(7)	–0.0073(7)
O(1)	2*i*	–0.0927(2)	–0.2305(2)	0.6186(2)	0.0465(9)	0.0353(8)	0.0201(7)	–0.0098(7)	–0.0024(6)	–0.0058(6)
O(2)	2*i*	–0.3706(2)	–0.2259(2)	0.4241(2)	0.0328(8)	0.0468(9)	0.0392(9)	–0.0206(7)	–0.0012(7)	0.0015(7)
O(3)	2*i*	–0.4543(2)	0.3415(2)	0.1771(2)	0.0364(9)	0.0310(8)	0.060(1)	–0.0041(7)	–0.0210(8)	–0.0028(7)
C(1)	2*i*	–0.0896(3)	–0.1352(2)	0.3443(2)	0.026(1)	0.0238(9)	0.0229(9)	–0.0087(8)	–0.0040(8)	–0.0046(7)
C(2)	2*i*	0.2148(3)	–0.1545(3)	0.2702(2)	0.023(1)	0.030(1)	0.037(1)	–0.0090(8)	–0.0066(8)	–0.0057(9)
C(3)	2*i*	0.1905(3)	–0.2159(3)	0.4248(2)	0.029(1)	0.032(1)	0.038(1)	–0.0083(9)	–0.0131(9)	–0.0055(9)
C(4)	2*i*	–0.2982(3)	–0.1104(3)	0.3515(2)	0.026(1)	0.035(1)	0.024(1)	–0.0108(9)	–0.0017(8)	–0.0093(8)
C(5)	2*i*	–0.4099(3)	0.0602(3)	0.2733(2)	0.022(1)	0.037(1)	0.037(1)	–0.0057(9)	–0.0073(8)	–0.0104(9)
C(6)	2*i*	–0.0784(4)	–0.4125(3)	0.6846(3)	0.047(1)	0.037(1)	0.030(1)	–0.013(1)	–0.005(1)	0.0020(9)
C(7)	2*i*	–0.3648(4)	0.4776(3)	0.1515(3)	0.061(2)	0.035(1)	0.065(2)	–0.015(1)	–0.024(1)	–0.004(1)

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