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The crystal structure of dichlorido(1,3-bis(2,6-dimethyl-phenyl)-1*H*-3*l*⁴-imidazol-2-yl)(2-methyl-4,5-dihydrooxazol- κ N)palladium(IV)-water (1/1), C₂₃H₂₉Cl₂N₃O₂Pd

Table 1: Data collection and handling.

Crystal:	Colourless prisma
Size:	0.18 × 0.15 × 0.12 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.98 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	25.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	31372, 4628, 0.052
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4106
$N(\text{param})_{\text{refined}}$:	286
Programs:	Bruker [1], SHELX [2]

conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

According to our very recent reported method, the title product was obtained in moderate yield [3]. Crystals suitable were grown in a mixture of ethyl acetate and petroleum ether.

Experimental details

All hydrogen atoms attached to C atoms were introduced using the HFIX command in the SHELXL program [2]. The C–H distances in CH₃ were restrained to 0.96 Å with U_{iso} values to be 1.5 $U_{\text{eq}}(\text{C})$. Vinylic and aromatic C–H distances were restrained to 0.93 Å with U_{iso} values to be 1.2 $U_{\text{eq}}(\text{C})$ [1].

Comment

Since the first isolation of stable free *N*-heterocyclic carbene (NHC) by Arduengo and co-workers in 1991 [4], the chemistry of NHC-metal complexes and their applications in organic synthesis were undoubtedly developed rapidly [5–10]. Crystal structures of various NHC complexes are important for the investigation of structure-activity relationship. Recently, the title product, together with its analogue, was found to be a good catalyst in the highly selective mono-Suzuki-Miyaura coupling of dichlorobenzenes with various aryl-boronic acids [1]. However, in the preliminary manuscript, only the crystal structure of one of the complexes was reported.

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Abstract

C₂₃H₂₉Cl₂N₃O₂Pd, monoclinic, $P2_1/c$ (no. 14), $a = 8.7287(6)$ Å, $b = 31.5431(18)$ Å, $c = 9.5605(6)$ Å, $\beta = 108.893(2)^\circ$, $V = 2490.5(3)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0438$, $wR_{\text{ref}}(F^2) = 0.1033$, $T = 293(2)$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
Pd1	0.62066(4)	0.35681(2)	0.34056(3)	0.04127(14)
Cl1	0.79525(17)	0.32181(5)	0.24565(14)	0.0722(4)
Cl2	0.44454(15)	0.38789(4)	0.44397(13)	0.0566(3)
N1	0.5045(5)	0.42131(11)	0.1048(4)	0.0527(9)
N2	0.4065(4)	0.36074(11)	0.0209(4)	0.0448(8)
N3	0.7627(5)	0.33732(13)	0.5498(4)	0.0543(9)
O1	0.9341(5)	0.34224(15)	0.7782(4)	0.0898(13)
C1	0.5021(5)	0.38007(13)	0.1438(4)	0.0405(9)
C2	0.4120(8)	0.42701(16)	−0.0406(5)	0.0724(16)
H2	0.3953	0.4525	−0.0925	0.087*
C3	0.3511(7)	0.38963(16)	−0.0933(5)	0.0649(14)
H3	0.2838	0.3839	−0.1889	0.078*
C4	0.3654(5)	0.31642(13)	0.0070(4)	0.0473(10)
C5	0.4427(7)	0.29075(18)	−0.0688(6)	0.0707(15)
C6	0.4022(11)	0.2481(2)	−0.0795(9)	0.111(3)
H6	0.4498	0.2301	−0.1307	0.134*
C7	0.2992(15)	0.2322(3)	−0.018(10)	0.137(5)
H7	0.2788	0.2032	−0.0262	0.164*
C8	0.2214(10)	0.2575(3)	0.0519(7)	0.107(3)
H8	0.1479	0.2455	0.0925	0.128*
C9	0.2514(6)	0.30167(19)	0.0649(5)	0.0663(14)
C10	0.1602(7)	0.3303(3)	0.1318(7)	0.098(2)
H10A	0.2287	0.3390	0.2279	0.147*
H10B	0.0674	0.3157	0.1408	0.147*
H10C	0.1253	0.3548	0.0700	0.147*
C11	0.5584(8)	0.3070(3)	−0.1386(7)	0.105(2)
H11A	0.6163	0.2838	−0.1626	0.157*
H11B	0.6336	0.3258	−0.0715	0.157*
H11C	0.5008	0.3221	−0.2273	0.157*
C12	0.5903(7)	0.45488(14)	0.1998(5)	0.0570(12)
C13	0.7525(8)	0.46033(16)	0.2224(6)	0.0699(14)
C14	0.8314(9)	0.49287(19)	0.3163(7)	0.0843(19)
H14	0.9419	0.4968	0.3353	0.101*
C15	0.7501(11)	0.51899(19)	0.3808(6)	0.094(2)
H15	0.8056	0.5404	0.4440	0.113*
C16	0.5873(10)	0.51415(17)	0.3535(6)	0.083(2)
H16	0.5332	0.5324	0.3981	0.099*
C17	0.5016(7)	0.48231(15)	0.2598(5)	0.0619(13)
C18	0.3220(8)	0.47824(19)	0.2252(7)	0.0847(18)
H18A	0.2682	0.4875	0.1256	0.127*
H18B	0.2878	0.4955	0.2924	0.127*
H18C	0.2950	0.4492	0.2353	0.127*
C19	0.8439(8)	0.4329(2)	0.1480(9)	0.101(2)
H19A	0.8435	0.4042	0.1804	0.151*
H19B	0.9535	0.4428	0.1733	0.151*
H19C	0.7931	0.4344	0.0428	0.151*
C20	0.8069(7)	0.29351(16)	0.5933(5)	0.0669(14)
H20A	0.8552	0.2800	0.5266	0.080*
H20B	0.7131	0.2772	0.5948	0.080*
C21	0.9273(8)	0.2976(2)	0.7459(6)	0.0861(18)
H21A	0.8920	0.2817	0.8168	0.103*
H21B	1.0327	0.2871	0.7483	0.103*
C22	0.8403(7)	0.36120(17)	0.6592(5)	0.0660(14)

Table 2 (continued)

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
C23	0.8390(10)	0.4081(2)	0.6670(8)	0.104(2)
H23A	0.9320	0.4192	0.6467	0.156*
H23B	0.8419	0.4167	0.7642	0.156*
H23C	0.7423	0.4188	0.5953	0.156*
O2	0.1089(7)	0.3699(2)	0.5198(7)	0.135(2)
H2A	0.0195	0.3600	0.4368	0.162*
H2B	0.2112	0.3753	0.5074	0.162*

In the crystal structure, one molecule of water is additionally present in the asymmetric unit. The same coordinate mode of the Pd center was obtained compared to the structure of its analogue [1]. Both structures almost share the same bond lengths Pd(1)—C(1) and Pd(1)—N(3) [1.970(4) vs 1.969(2) Å and 2.074(3) vs 2.078(2) Å, respectively]. Both Pd centers showed slightly distorted square-planar geometry with four ligands. However, it seems that the previous reported one was more planar than the current one. The bond angles around the Pd center are more close to 180° in the previous reported one [C(1)—Pd(1)—N(3): 177.54(9) vs 174.19(16)°; Cl(1)—Pd(1)—Cl(2): 178.58(3) vs 176.23°]. In general geometric parameters are as expected [11].

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