Jian Yang*

The crystal structure of dichlorido(1,3-dimesityl-1*H*-3 λ^4 -imidazol-2-yl)(morpholine- κ N)palladium(IV), C₂₅H₃₃Cl₂N₃OPd

Table 1: Data collection and handling.

Crystal:	Yellow prismatic
Size:	0.18 × 0.13 × 0.10 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.97 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	26.0°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	22323, 4949, 0.038
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4676
$N(\text{param})_{\text{refined}}$:	300
Programs:	SHELX [1], Bruker [2]

The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

By Shao and co-worker's pioneering work, the title compound was obtained in 80% isolated yields after flash column chromatography [3]. Crystals suitable for X-ray diffraction were obtained by slow evaporation of the mixed solvents of dichloromethane and ethyl acetate.

Experimental details

All hydrogen atoms attached to C atoms were introduced using the HFIX command in the SHELXL program [1]. The C–H distances in CH₂ were restrained to 0.99 Å with U_{iso} values to be 1.2 U_{eq} (C). The C–H distances in CH₃ were restrained to 0.98 Å with U_{iso} values to be 1.5 U_{eq} (C). All other vinylic and aromatic C–H distances were restrained to 0.95 Å with U_{iso} values to be 1.2 U_{eq} (C) [1].

Comment

In 2012, Shao and co-workers found that the *iso*-propyl analogue of the title compound is the active catalytic species for the Suzuki-Miyaura coupling of aryl sulfonates [4]. In the next year, the same group reported the facile synthesis of the title compound and its analogue and showed them to be efficient catalysts in the Suzuki-Miyaura coupling of aryl sulfonates with arylboronic acids. However, only crystal structure of the *iso*-propyl analogue of the title compound was reported

<https://doi.org/10.1515/ncrs-2018-0131>

Received April 17, 2018; accepted July 19, 2018; available online August 15, 2018

Abstract

C₂₅H₃₃Cl₂N₃OPd, triclinic, $P\bar{1}$ (no. 2), $a = 10.2439(9)$ Å, $b = 11.4323(9)$ Å, $c = 11.9773(11)$ Å, $\alpha = 93.043(3)^\circ$, $\beta = 94.892(3)^\circ$, $\gamma = 114.364(2)^\circ$, $V = 1267.07(19)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0212$, $wR_{\text{ref}}(F^2) = 0.0528$, $T = 173(2)$ K.

CCDC no.: 1856821

*Corresponding author: Jian Yang, College of Resources and Environment, Hunan Agricultural University, Hunan Province 410128, People's Republic of China, e-mail: yangjian188@hunau.edu.cn

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	0.57010(2)	0.35190(2)	0.21433(2)	0.01767(7)
H3	0.391(2)	0.3674(19)	0.0794(16)	0.025(6)*
Cl1	0.66752(5)	0.44321(5)	0.05680(4)	0.03315(12)
Cl2	0.46244(5)	0.25397(5)	0.36544(4)	0.02939(11)
O1	0.06454(14)	0.17760(14)	0.06020(13)	0.0351(3)
N1	0.80176(15)	0.27759(14)	0.30940(12)	0.0191(3)
N2	0.88048(15)	0.48325(14)	0.31415(13)	0.0193(3)
N3	0.36886(16)	0.31930(16)	0.12718(14)	0.0233(3)
C1	0.76030(18)	0.37259(17)	0.28440(14)	0.0186(4)
C2	0.94546(19)	0.32908(18)	0.35467(16)	0.0228(4)
H2	0.9988	0.2821	0.3788	0.027*
C3	0.99450(19)	0.45704(18)	0.35784(16)	0.0227(4)
H3A	1.0894	0.5186	0.3848	0.027*
C4	0.89782(19)	0.61480(17)	0.30710(15)	0.0199(4)
C5	0.99278(19)	0.68826(18)	0.23455(16)	0.0232(4)
C6	1.0232(2)	0.81870(19)	0.23823(17)	0.0279(4)
H6	1.0879	0.8708	0.1900	0.034*
C7	0.9616(2)	0.87509(18)	0.31049(18)	0.0293(4)
C8	0.8652(2)	0.79791(19)	0.37862(17)	0.0276(4)
H8	0.8205	0.8356	0.4263	0.033*
C9	0.8313(2)	0.66617(18)	0.37961(16)	0.0232(4)
C10	0.7273(2)	0.5875(2)	0.45655(17)	0.0287(4)
H10A	0.6318	0.5400	0.4139	0.043*
H10B	0.7211	0.6449	0.5176	0.043*
H10C	0.7612	0.5264	0.4883	0.043*
C11	1.0604(2)	0.6301(2)	0.15393(17)	0.0298(4)
H11A	1.1345	0.6110	0.1961	0.045*
H11B	1.1047	0.6913	0.0993	0.045*
H11C	0.9860	0.5502	0.1141	0.045*
C12	1.0001(3)	1.0179(2)	0.3156(2)	0.0434(6)
H12A	1.1032	1.0663	0.3414	0.065*
H12B	0.9435	1.0397	0.3681	0.065*
H12C	0.9788	1.0404	0.2405	0.065*
C13	0.71221(18)	0.14014(17)	0.30020(15)	0.0192(4)
C14	0.66583(19)	0.06977(18)	0.19467(16)	0.0221(4)
C15	0.5868(2)	−0.06326(19)	0.19288(17)	0.0274(4)
H15	0.5506	−0.1133	0.1221	0.033*
C16	0.5592(2)	−0.12519(18)	0.28998(18)	0.0279(4)
C17	0.6082(2)	−0.05154(19)	0.39288(17)	0.0271(4)
H17	0.5896	−0.0931	0.4601	0.033*
C18	0.68414(19)	0.08195(18)	0.40033(16)	0.0222(4)
C19	0.7339(2)	0.1593(2)	0.51387(16)	0.0297(4)
H19A	0.8390	0.1916	0.5295	0.045*
H19B	0.6881	0.1042	0.5720	0.045*
H19C	0.7070	0.2322	0.5138	0.045*
C20	0.7036(2)	0.1306(2)	0.08676(16)	0.0292(4)

Table 2 (continued)

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
H20A	0.6859	0.2086	0.0874	0.044*
H20B	0.6437	0.0694	0.0228	0.044*
H20C	0.8056	0.1534	0.0798	0.044*
C21	0.4801(3)	−0.2705(2)	0.2832(2)	0.0431(6)
H21A	0.4001	−0.3005	0.2223	0.065*
H21B	0.4422	−0.2959	0.3548	0.065*
H21C	0.5468	−0.3094	0.2680	0.065*
C22	0.2601(2)	0.34574(18)	0.18372(17)	0.0254(4)
H22A	0.2997	0.4387	0.2102	0.030*
H22B	0.2381	0.2963	0.2503	0.030*
C23	0.1221(2)	0.3090(2)	0.10485(18)	0.0302(4)
H23A	0.0498	0.3238	0.1462	0.036*
H23B	0.1423	0.3647	0.0422	0.036*
C24	0.1667(2)	0.1541(3)	0.0001(2)	0.0462(6)
H24A	0.1892	0.2088	−0.0633	0.055*
H24B	0.1249	0.0627	−0.0315	0.055*
C25	0.3040(2)	0.1838(2)	0.07701(19)	0.0354(5)
H25A	0.2822	0.1255	0.1378	0.043*
H25B	0.3740	0.1678	0.0336	0.043*

therein. Therefore, we think it necessary to report the crystal structure of the title compound in this case to understand more information about such compounds.

The structure of the title complex is very similar to its iso-propyl analogue. The Pd—C_{carbene} and Pd—N bond lengths of the title compound are 1.9688(17) and 2.1062(15) Å, respectively. Generally all geometric parameters are in perfect accordance with the literature [5].

Acknowledgements: I acknowledge Hunan Agricultural University for its financial support for this project.

References

- Sheldrick, G. M.: A short history of SHELX. *Acta Crystallogr.* **A64** (2008) 112–122.
- Bruker. SADABS, Version 2.03, SAINT, Version 6.02. Bruker AXS Inc. Madison, WI, USA (2002).
- Wang, Z.-Y.; Ma, Q.-N.; Li, R.-H.; Shao, L.-X.: Palladium-catalyzed Suzuki-Miyaura coupling of aryl sulfamates with arylboronic acids. *Org. Biomol. Chem.* **11** (2013) 7899–7906.
- Wang, Z.-Y.; Chen, G.-Q.; Shao, L.-X.: N-Heterocyclic carbene-palladium(II)-1-methylimidazole complex-catalyzed Suzuki-Miyaura coupling of aryl sulfonates with arylboronic acids. *J. Org. Chem.* **77** (2012) 6608–6614.
- Liu, F.; Li, G.-X.: The crystal structure of dichlorido (1,3-dimesityl-1*H*-3*λ*⁴-imidazol-2-yl)(isoquinoline-κN)palladium(IV) – ethylacetate (1/1), C₃₄H₃₉Cl₂N₃O₂Pd. *Z. Kristallogr. NCS* **231** (2016) 1143–1144.