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The crystal structure of dichlorido-(2-((4-phenyl-2*H*-1,2,3-triazol-2-yl)methyl)pyridine- $\kappa^2 N, N'$) palladium(II), $C_{14}H_{12}Cl_2N_4Pd$

Table 1: Data collection and handling.

Crystal:	Red block
Size:	0.12 × 0.11 × 0.10 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	1.57 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	32.2°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	16,188, 5025, 0.030
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4181
$N(\text{param})_{\text{refined}}$:	190
Programs:	Bruker [1], SHELX [2–4], Olex2 [5], PLATON [6]

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Abstract

$C_{14}H_{12}Cl_2N_4Pd$, monoclinic, $P2_1/c$ (no. 14), $a = 11.990(3)$ Å, $b = 13.027(3)$ Å, $c = 9.730(2)$ Å, $\beta = 92.163(4)^\circ$, $V = 1518.7(6)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0290$, $wR_{\text{ref}}(F^2) = 0.0856$, $T = 296(2)$ K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

$Pd(CH_3CN)_2Cl_2$ (0.100 g, 0.345 mmol) was placed in a 25 mL round-bottomed flask, and 10 mL of dry CH_3CN was added to form a red brown solution. Then, 2-((4-phenyl-2*H*-1,2,3-triazol-2-yl)methyl) pyridine (0.082 g, 0.345 mmol)

was added. Reaction was allowed to stir at room temperature for 2 min. The solution was evaporated completely under reduced pressure and the obtained residue was redissolved in 5 mL of dichloromethane. Diethyl ether was added with stirring to induce precipitation. The solution was discarded by filtration and the precipitate was further washed with diethyl ether (5 mL). Finally, it was dried under vacuum to afford the title compound as yellow powder. Red brown crystals were grown through a slow evaporation of a diethyl ether/*N,N*-dimethyl-formamide ($V/V = 1:5$) solution for two weeks.

Experimental details

Hydrogen atoms bonded to carbon atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms with $C-H = 0.93$ Å (aromatic), 0.97 Å (methylene) and $U_{\text{iso}}H = 1.2U_{\text{eq}}C$.

Comment

1,2,3-Triazole, as an important class of nitrogen-containing heterocyclic compounds, has a high electron cloud density to be used as a coordination site [7–9]. 1,2,3-Triazole compounds could serve as efficient ligands for Pd-catalyzed Suzuki reaction [10]. In this paper, a new 1,2,3-triazole palladium complex is described.

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

Atom	x	y	z	U _{iso} */U _{eq}
Pd1	0.60704 (2)	−0.07421 (2)	0.08895 (2)	0.02879 (6)
C1	0.6630 (2)	0.06725 (17)	0.3230 (2)	0.0344 (4)
C2	0.6862 (2)	0.0932 (2)	0.4586 (3)	0.0449 (6)
H2	0.7155	0.1574	0.4806	0.054*
C3	0.6660 (3)	0.0236 (2)	0.5610 (2)	0.0524 (7)
H3	0.6801	0.0406	0.6529	0.063*
C4	0.6246 (2)	−0.0711 (2)	0.5250 (3)	0.0474 (6)
H4	0.6115	−0.1197	0.5925	0.057*
C5	0.6026 (2)	−0.09382 (19)	0.3890 (2)	0.0388 (5)
H5	0.5739	−0.1580	0.3653	0.047*
C6	0.6807 (2)	0.14282 (17)	0.2094 (2)	0.0370 (5)
H6A	0.6093	0.1590	0.1644	0.044*
H6B	0.7112	0.2058	0.2484	0.044*
C7	0.81577 (19)	0.00852 (18)	−0.0483 (2)	0.0362 (5)
H7	0.8267	−0.0460	−0.1080	0.043*
C8	0.87645 (19)	0.0987 (2)	−0.0425 (2)	0.0377 (5)
C9	0.9692 (2)	0.1327 (2)	−0.1265 (3)	0.0441 (6)
C10	1.0053 (3)	0.0721 (2)	−0.2321 (3)	0.0551 (7)
H10	0.9702	0.0099	−0.2518	0.066*
C11	1.0942 (3)	0.1041 (3)	−0.3092 (4)	0.0687 (9)
H11	1.1190	0.0628	−0.3798	0.082*
C12	1.1451 (3)	0.1958 (3)	−0.2816 (4)	0.0733 (10)
H12	1.2035	0.2176	−0.3348	0.088*
C13	1.1109 (3)	0.2552 (3)	−0.1768 (4)	0.0796 (12)
H13	1.1475	0.3166	−0.1562	0.096*
C14	1.0213 (3)	0.2246 (3)	−0.1001 (4)	0.0641 (8)
H14	0.9965	0.2668	−0.0304	0.077*
Cl1	0.45269 (5)	−0.16948 (5)	0.12924 (6)	0.04411 (14)
Cl2	0.60899 (5)	−0.13591 (5)	−0.12937 (6)	0.04178 (13)
N1	0.62173 (15)	−0.02549 (14)	0.28911 (17)	0.0314 (4)
N2	0.73950 (15)	0.01351 (14)	0.04603 (18)	0.0320 (4)
N3	0.75611 (16)	0.10360 (15)	0.10723 (19)	0.0339 (4)
N4	0.83624 (17)	0.15799 (16)	0.0569 (2)	0.0399 (4)

The title compound crystallizes in the monoclinic $P2_1/c$ space group with one title complex in the asymmetric unit. The palladium (II) is four-coordinated by two chlorine atoms and two nitrogen atoms. One of the nitrogen atoms is derived from the 1,2,3-triazole ring and the other derived from pyridine moiety of the organic ligand [11]. In each complex, the Pd–N bond lengths range from 2.0131(18) to 2.0496(18) Å and the bond angles around each Pd1 center are in the range 88.33(5)–177.27(6)°, which exhibit a slight

distortion. The structure of title compound is further stabilized by intermolecular C–H···Cl hydrogen bonds.

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References

1. Bruker. SMART and SAINT; Bruker AXS Inc.: Madison, WI, USA, 2003.
2. Sheldrick G. M. SHELXTL integrated space-group and crystal structure determination. *Acta Crystallogr.* 2015, *A71*, 3–8.
3. Sheldrick G. M. Crystal structure refinement with SHELXL. *Acta Crystallogr.* 2015, *C71*, 3–8.
4. Sheldrick G. M. A short history of SHELX. *Acta Crystallogr.* 2008, *A64*, 112–122.
5. Dolomanov O. V., Bourhis L. J., Gildea R. J., Howard J. A. K., Puschmann H. OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* 2009, *42*, 339–341.
6. Spek A. L. Structure validation in chemical crystallography. *Acta Crystallogr.* 2009, *D65*, 148–155.
7. Choubey B., Radhakrishna L., Mague J. T., Balakrishna M. S. Two triazole-based phosphine ligands prepared via temperature-mediated Li/H exchange: CuI and AuI complexes and structural studies. *Inorg. Chem.* 2016, *55*, 8514–8526.
8. Manbeck G. F., Brennessel W. W., Evans C. M., Eisenberg R. Tetranuclear copper(I) iodide complexes of chelating bis(1-benzyl-1H-1,2,3-triazole) ligands: structural characterization and solid state photoluminescence. *Inorg. Chem.* 2010, *49*, 2834–2843.
9. Suijkerbuijk B. M. J. M., Aerts B. N. H., Dijkstra H. P., Lutz M., Spek A. L., van Koten G., Klein Gebbink R. J. M. “Click” 1,2,3-triazoles as tunable ligands for late transition metal complexes. *Dalton Trans.* 2007, *13*, 1273–1276.
10. Yan Q., Zheng L., Li M., Chen Y. N. S-chelating triazole- thioether ligand for highly efficient palladium-catalyzed Suzuki reaction. *J. Catal.* 2019, *376*, 101–105.
11. Chen Y., Wu J., Ma S., Zhou S., Meng X., Jia L., Pan Z. Syntheses, structures and properties of Zn(II) and Cu(II) complexes based on N2-2-methylenepyridinyl 1,2,3-triazole ligand. *J. Mol. Struct.* 2015, *1089*, 1–8.