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Crystal structure of *cyclo*-[tetrachlorido-bis(μ_2 -*p*-xylylenediamine- $\kappa^2 N:N'$)dipalladium(II)] dimethyl sulfoxide solvate, $C_{20}H_{36}Cl_4N_4O_2Pd_2S_2$

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.13 × 0.10 × 0.05 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.74 mm ⁻¹
Diffractometer, scan mode:	PHOTON 100 CMOS, φ and ω
$\theta_{\max}$, completeness:	28.3°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	92873, 3644, 0.096
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3037
$N(\text{param})_{\text{refined}}$:	156
Programs:	Bruker [1], SHELX [2], ORTEP-3 [3], Platon [4]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Pd1	0.14995(2)	0.34518(2)	0.22290(5)	0.02295(8)
Cl1	0.24240(9)	0.43944(9)	0.23687(7)	0.0438(4)
Cl2	0.06969(10)	0.24040(9)	0.21084(7)	0.0502(5)
N1	0.1408(3)	0.3376(3)	0.3256(3)	0.0272(14)
H1A	0.1611	0.2921	0.3385	0.033*
H1B	0.1703	0.3751	0.3439	0.033*
N2	0.1579(3)	0.3496(3)	0.1197(4)	0.0282(15)
H2A	0.2039	0.3296	0.1078	0.034*
H2B	0.1213	0.3182	0.1026	0.034*
C1	0.0625(3)	0.3438(3)	0.3558(3)	0.0321(14)
H1C	0.0644	0.3257	0.4029	0.038*
H1D	0.0272	0.3099	0.3309	0.038*
C2	0.0311(3)	0.4246(3)	0.3547(3)	0.0252(12)
C3	0.0157(3)	0.4628(3)	0.4143(2)	0.0334(12)
H3	0.0265	0.4384	0.4559	0.040*
C4	0.0148(3)	0.4633(3)	0.2940(2)	0.0280(11)
H4	0.0242	0.4384	0.2525	0.034*
C5	0.1502(3)	0.4254(3)	0.0858(3)	0.0328(13)
H5A	0.1596	0.4187	0.0372	0.039*
H5B	0.1900	0.4603	0.1035	0.039*
C6	0.0724(3)	0.4623(3)	0.0955(3)	0.0268(12)
C7	0.0040(3)	0.4204(3)	0.0967(3)	0.0313(11)
H7	0.0059	0.3662	0.0972	0.038*
C8	0.0672(3)	0.5425(3)	0.0971(3)	0.0310(11)
H8	0.1127	0.5721	0.0985	0.037*
S1	0.25896(8)	0.13619(7)	0.31097(7)	0.0367(3)
O1	0.2463(3)	0.21404(17)	0.3429(3)	0.0403(8)

<https://doi.org/10.1515/ncrs-2019-0867>

Received November 27, 2019; accepted January 10, 2020; available online January 29, 2020

Abstract

$C_{20}H_{36}Cl_4N_4O_2Pd_2S_2$, tetragonal, $I4_1cd$ (no. 110), $a = 17.3126(19)$ Å, $c = 19.725(2)$ Å, $V = 5912.1(14)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0284$, $wR_{\text{ref}}(F^2) = 0.0536$, $T = 223(2)$ K.

CCDC no.: 1976493

Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

To a solution of Na_2PdCl_4 (0.2943 g; 1.000 mmol) in MeOH (30 mL) was added *p*-xylylenediamine (0.1402 g, 1.029 mmol) and stirred for 3 h at room temperature. The formed precipitate was separated by filtration, washed with H₂O and

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Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C9	0.3248(4)	0.0893(4)	0.3655(3)	0.0575(18)
H9A	0.3672	0.1239	0.3759	0.086*
H9B	0.3446	0.0433	0.3434	0.086*
H9C	0.2986	0.0748	0.4071	0.086*
C10	0.3231(5)	0.1524(4)	0.2421(3)	0.077(2)
H10A	0.2978	0.1843	0.2083	0.115*
H10B	0.3375	0.1033	0.2220	0.115*
H10C	0.3691	0.1786	0.2584	0.115*

acetone, and dried at 50 °C, to give a yellow powder (0.2875 g). Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation from a dimethyl sulfoxide (DMSO) solution at 90 °C.

Experimental details

Hydrogen atoms were positioned geometrically and allowed to ride on their parent atoms with $d(\text{N}-\text{H}) = 0.90 \text{ \AA}$, $d(\text{C}-\text{H}) = 0.98, 0.94 \text{ or } 0.97 \text{ \AA}$ and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N}, \text{C})$ or $1.5U_{\text{eq}}(\text{methyl C})$ with the help of the SHELXL program (AFIX 23, 43 or 137 options) [2]. The highest peak ($0.43 \text{ e \AA}^{-3}$) and the deepest hole ($-0.30 \text{ e \AA}^{-3}$) in the difference Fourier map are located $0.55 \text{ \AA}$ and $1.39 \text{ \AA}$ from the atoms C2 and C8, respectively.

Comment

The crystal structures of the related *p*-xylylenediamine-bridged complexes $[\text{M}(\mu_3\text{-SO}_4)(\mu_2\text{-pxd})]_n$ ($\text{M} = \text{Zn}, \text{Cd}$; $\text{pxd} = p\text{-xylylenediamine}$) [5] and $[\text{Zn}_2(\mu_3\text{-S})_2(\mu_2\text{-pxd})]_n$ [6] have been determined previously by X-ray powder diffraction techniques. Furthermore there is a single crystal structure determination of a Cd(II) complex [7].

The title complex has a cyclic dinuclear structure, in which two *trans*-chlorido-Pd(II) units $[\text{PdCl}_2]$ are linked by two pxd ligands. In the crystal, the benzene rings are positioned exactly perpendicular to each other. The complex is disposed about a twofold rotation axis running in the [001] direction passing through the centroid of one benzene ring, and bisecting the other benzene ring; the asymmetric unit

contains one half of the compound (see the figure, the asymmetric unit is labelled). In the complex, each Pd(II) ion is four-coordinated in a slightly distorted square-planar environment by two Cl^- ligands and two mutually *trans*-positioned N atoms derived from two bridging bidentate pxd ligands. The Pd–Cl and Pd–N bond lengths are almost equal with $d(\text{Pd}-\text{Cl}) = 2.2974(15)$ and $2.3023(14) \text{ \AA}$, and $d(\text{Pd}-\text{N}) = 2.037(7)$ and $2.042(7) \text{ \AA}$, respectively. In the crystal structure, the complexes and solvent DMSO molecules are linked by $\text{N}-\text{H} \cdots \text{O}$, $\text{N}-\text{H} \cdots \text{Cl}$, $\text{C}-\text{H} \cdots \text{Cl}$ and $\text{C}-\text{H} \cdots \text{O}$ hydrogen bonds with distances of $2.834(6)$ – $3.558(7) \text{ \AA}$ between the donor and acceptor atoms, forming a three-dimensional network [4].

Acknowledgements: This research was supported by Basic Science Research Program through the National Research Foundation of Korea (NRF) funded by the Ministry of Education (grant number: 2018R1D1A1B07050550). The author thanks the KBSI, Seoul Center, for the X-ray data collection.

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