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Crystal structure of 1,1'-(1,3-phenylenebis(methylene))bis(pyridin-1-ium) bis(1,2-dicyanoethene-1,2-dithiolato- $\kappa^2S:S$) palladium(II), $C_{26}H_{18}N_6PdS_4$

Table 1: Data collection and handling.

Crystal:	Brown block
Size:	0.24 × 0.22 × 0.20 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.04 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX II
$\theta_{\max}$, completeness:	27.8°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	8293, 3130, 0.022
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2764
$N(\text{param})_{\text{refined}}$:	170
Programs:	SHELX [1], Bruker [2]

Source of material

All reagents and chemicals were purchased from commercial sources and used without further purification. The starting materials disodium maleonitriledithiolate and 1,3-bis(methylpyridinium benzene) bromide were synthesized following the literature procedures [3, 4]. An aqueous solution (10 mL) of 1,3-bis(methylpyridinium benzene) bromide (0.0842 g, 0.2 mmol) was added slowly to an aqueous solution (20 mL) of disodium maleonitriledithiolate (0.0741 g, 0.4 mmol) and K_2PdCl_4 (0.0982 g, 0.3 mmol), and the mixture was stirred at room temperature for several minutes. A brown precipitate was filtered off, washed by water and dried under vacuum. The precipitate was dissolved in dimethylformamide with ether diffusion. Two weeks later brown crystals were obtained.

Experimental details

Absorption corrections were applied using the multi-scan method. Hydrogen atoms were located in difference electron density maps, and treated as riding atoms. The U_{iso} values of the hydrogen atoms were set to $1.2U_{\text{eq}}(C)$.

Comment

Complexes have been extensively studied for a long time, since potentially they could be candidates for magne

<https://doi.org/10.1515/ncrs-2021-0292>

Received July 17, 2021; accepted August 20, 2021;

published online October 1, 2021

Abstract

$C_{26}H_{18}N_6PdS_4$, monoclinic, $C2/c$ (no. 15), $a = 16.658(2)$ Å, $b = 10.398(3)$ Å, $c = 17.284(2)$ Å, $\beta = 116.928(3)^\circ$, $V = 2669.2(9)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0330$, $wR_{\text{ref}}(F^2) = 0.0912$, $T = 293(2)$ K.

CCDC no.: 2104562

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.

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$).

Atom	x	y	z	U_{iso}^*/U_{eq}
C1	0.12172 (17)	0.6429 (2)	0.46994 (16)	0.0407 (5)
C2	0.17154 (16)	0.5313 (2)	0.47022 (15)	0.0371 (5)
C3	0.22182 (16)	0.5315 (2)	0.42544 (15)	0.0366 (5)
C4	0.22507 (17)	0.6431 (2)	0.37937 (16)	0.0396 (5)
C5	0.000000	0.6132 (3)	0.250000	0.0443 (8)
H5	−0.000001	0.702600	0.249999	0.053*
C6	0.04345 (17)	0.5482 (2)	0.21186 (16)	0.0392 (5)
H6	0.073089	0.592816	0.185729	0.047*
C7	0.04323 (17)	0.4165 (2)	0.21221 (16)	0.0380 (5)
C8	0.000000	0.3518 (3)	0.250000	0.0416 (8)
H8	−0.000001	0.262399	0.249999	0.050*
C9	0.0878 (2)	0.3488 (2)	0.16342 (19)	0.0478 (6)
H9A	0.055707	0.370588	0.102325	0.057*
H9B	0.149166	0.379364	0.185013	0.057*
C10	0.02345 (19)	0.1362 (3)	0.11344 (17)	0.0470 (6)
H10	−0.019892	0.175013	0.063476	0.056*
C11	0.0204 (2)	0.0086 (3)	0.1256 (2)	0.0549 (7)
H11	−0.025135	−0.040860	0.083890	0.066*
C12	0.0846 (2)	−0.0484 (2)	0.1994 (2)	0.0592 (7)
H12	0.082759	−0.136544	0.207764	0.071*
C13	0.1496 (2)	0.0233 (3)	0.2592 (2)	0.0532 (7)
H13	0.193265	−0.014487	0.309464	0.064*
C14	0.15167 (18)	0.1517 (2)	0.24617 (16)	0.0449 (6)
H14	0.196659	0.202211	0.287649	0.054*
N1	0.07857 (18)	0.7308 (2)	0.46888 (16)	0.0470 (6)
N2	0.22659 (17)	0.73123 (19)	0.33957 (16)	0.0461 (6)
N3	0.08861 (15)	0.2060 (2)	0.17322 (14)	0.0392 (5)
Pd1	0.250000	0.250000	0.500000	0.03578 (11)
S1	0.16297 (4)	0.39845 (5)	0.52664 (4)	0.03943 (16)
S2	0.28296 (4)	0.39979 (5)	0.42148 (4)	0.03920 (16)

tism, luminescence and catalysis as well as pharmaceutical aspect, etc. [5–9]. Among these numerous complexes those with the maleonitriledithiolate (mnt) ligand have been extensively studied. Varieties of maleonitriledithiolate complexes have shown unusual optical, electrical, magnetic properties and so on [10–14]. This contribution is part of our present project on the design and synthesis of complexes based on the mnt ligand [15, 16]. Herein, we choose 1,3-bis(methylpyridinium benzene) as organic divalent cation $[1,3\text{-BMPB}]^{2+}$, to stabilize the anionic palladium(II) complex. X-ray crystallography analyses reveal that the title complex shows 3D framework linked through extensive hydrogen bonding interactions.

The asymmetric contains one half of the $[\text{Pd}(\text{mnt})_2]^{2-}$ dianion and of the 1,3-bis(methylpyridinium benzene) dication (see the Figure). The Pd(II) is coordinated by four S atoms of two maleonitriledithiolate ligands, and exhibits

the expected square-planar coordination geometry. The Pd1–S bond lengths and S–Pd1–S bond angles are in 2.2900(7)–2.3025(7) Å and 89.76(3)–180°, respectively. All bond lengths and bond angles are consistent with the reported complexes [17]. In comparison with a reported complex [15], the anions and cations do not form completely separated columns but cross each other. This difference may be due to cation structure hindrance. Some weak C–H⋯N interactions are observed between anions and cations [18].

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: This study was financially support by Scientific and Technological Research Projects of Henan Province (182102311077).

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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