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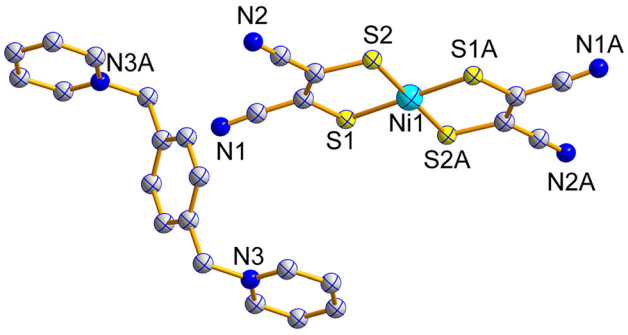
Crystal structure of 1,4-bis(methylpyridinium benzene) bis(1,2-dicyanoethene-1,2-dithiolato-κ$_2$*S*:*S*)nickel(II), $C_{26}H_{18}N_6NiS_4$



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Abstract

$C_{26}H_{18}N_6NiS_4$, triclinic, $P\bar{1}$ (no. 2), a = 8.2947(4) Å, b = 8.9964(5) Å, c = 9.4210(5) Å, α = 70.988(1)°, β = 79.971(1)°, γ = 82.567(1)°, V = 652.52(6) Å^3, Z = 1, $R_{gt}(F)$ = 0.0340, $wR_{ref}(F^2)$ = 0.0643, T = 296(2) K.

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Table 1: Data collection and handling.

Crystal:	Red block
Size:	0.18 × 0.16 × 0.16 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	1.09 mm^{-1}
Diffractometer, scan mode:	Bruker CCD APEX II,
θ_{max}, completeness:	25.0°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	3709, 2285, 0.046
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 1795
$N(param)_{refined}$:	169
Programs:	SHELX [1], Bruker [2]

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

Source of material

All reagents and chemicals were purchased from commercial sources and used without further purification. The starting materials disodium maleonitriledithiolate and 1,4-bis(methylpyridinium benzene bromide) were synthesized following the literature procedures [3, 4]. An aqueous solution (10 mL) of 1,4-bis(methylpyridinium benzene bromide (0.0842 g, 0.2 mmol) was added slowly to an

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
NI1	1.000000	0.000000	0.000000	0.03773 (15)
N1	0.9928 (3)	0.3464 (3)	0.3896 (3)	0.0630 (7)
N2	0.6817 (3)	−0.0201 (3)	0.5746 (3)	0.0620 (7)
N3	0.3109 (3)	0.6872 (3)	0.1478 (2)	0.0428 (6)
S1	1.08528 (9)	0.16969 (9)	0.08286 (7)	0.0507 (2)
S2	0.84212 (10)	−0.09661 (8)	0.21113 (7)	0.0485 (2)
C1	0.9665 (3)	0.1409 (3)	0.2596 (3)	0.0419 (7)
C2	0.9798 (3)	0.2525 (4)	0.3359 (3)	0.0463 (7)
C3	0.8622 (3)	0.0266 (3)	0.3145 (3)	0.0399 (7)
C4	0.7618 (3)	0.0017 (3)	0.4591 (3)	0.0439 (7)
C5	0.2385 (4)	0.5943 (4)	0.0995 (3)	0.0579 (8)
H5	0.157649	0.532901	0.165014	0.070*
C6	0.2801 (5)	0.5863 (4)	−0.0459 (4)	0.0724 (10)
H6	0.229756	0.518930	−0.078264	0.087*
C7	0.3958 (5)	0.6787 (5)	−0.1409 (4)	0.0778 (12)
H7	0.424905	0.676181	−0.240025	0.093*
C8	0.4695 (4)	0.7751 (5)	−0.0907 (3)	0.0775 (12)
H8	0.548828	0.838877	−0.155504	0.093*
C9	0.4265 (4)	0.7777 (4)	0.0541 (3)	0.0617 (9)
H9	0.477593	0.842725	0.088758	0.074*
C10	0.2645 (4)	0.6918 (4)	0.3075 (3)	0.0541 (8)
H10A	0.156406	0.653663	0.347180	0.065*
H10B	0.259375	0.799967	0.308079	0.065*
C11	0.3863 (3)	0.5925 (3)	0.4078 (3)	0.0409 (7)
C12	0.3662 (4)	0.4350 (3)	0.4834 (3)	0.0525 (8)
H12	0.275454	0.390074	0.473794	0.063*
C13	0.5202 (4)	0.6561 (3)	0.4270 (3)	0.0516 (8)
H13	0.534207	0.762540	0.378551	0.062*

aqueous solution (15 mL) of disodium maleonitriledithiolate (0.0741 g, 0.4 mmol) and $NiCl_2 \cdot 6H_2O$ (0.0476 g, 0.2 mmol), and the mixture was stirred at room temperature for several minutes. A red precipitate was filtered off, washed by water and dried under vacuum. The precipitate was dissolved in DMF with ether diffusion. Two weeks later red crystals were obtained.

Experimental details

Absorption corrections were applied by using multi-scan program. 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 1.2 U_{eq}(C).

Comment

The maleonitriledithiolate (mnt) ligand has been extensively studied in the past decades [5–10]. Considerable interests have been focused on maleonitriledithiolate complexes because of their special structures and diversity in physical and chemical properties. The ligand possesses planar conjugate structures and high delocalization, and it shows various weak interactions. Herein, we choose 1,4-bis(methylpyridinium benzene) as organic divalent cation $[1,4\text{-BMPB}]^{2+}$, to generate the new nickel(II) complex.

The title complex crystallizes in the triclinic space group $P\bar{1}$, with one half of $[Ni(mnt)_2]^{2-}$ dianion and one half of 1,4-bis(methylpyridinium benzene) dication in the asymmetric unit (see the Figure). Each Ni(II) ion is coordinated by four sulfur atoms, and exhibits quasi-square planar coordination geometry. The Ni1–S bond lengths and S–Ni1–S bond angles are in 2.1651(6)–2.1652(7) Å and 87.51(3)-180.0°, respectively, which are comparable to these reported for another $[Ni(mnt)_2]^{2-}$ complex [11]. The anions and cations are forming segregated columns. The C–H···N and C–H···π interactions are observed between anions and cations [12]. The weak intermolecular interactions lead to a 3D supramolecular structure.

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

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