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Crystal structure of tetraqua-bis(μ_2 -5-aminoisophthalato- $\kappa^3 N:O,O'$)-bis(4,4'-dipyridylsulfide- $\kappa^1 N$)dizinc(II), $C_{36}H_{34}N_6O_{12}S_2Ni_2$

Table 1: Data collection and handling.

Crystal:	Green block
Size:	0.29 × 0.25 × 0.25 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	1.18 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω
$\theta_{\max}$, completeness:	25°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	15078, 4181, 0.092
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2929
$N(\text{param})_{\text{refined}}$:	264
Programs:	CrysAlis ^{PRO} [1], SHELX [2, 3]

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Abstract

$C_{36}H_{34}N_6O_{12}S_2Ni_2$, monoclinic, $C2/c$ (no. 15), $a = 29.642(4)$ Å, $b = 6.2241(4)$ Å, $c = 21.667(2)$ Å, $\beta = 108.592(13)^\circ$, $V = 3788.9(7)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0907$, $wR_{\text{ref}}(F^2) = 0.1605$, $T = 293(2)$ 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

5-Aminoisophthalic acid (AIPH₂) (0.02 g, 0.1 mmol), 4,4'-dipyridylsulfide (dps; 0.02 g, 0.1 mmol) and nickel(II) acetate tetrahydrate (0.03 g, 0.1 mmol) were added to water (7 mL) in a Teflon-lined stainless steel reactor with a small amount of a base. The mixture was heated at 393 K for 3 d, and then slowly

cooled down to room temperature. Green crystals of the title compound were obtained.

Experimental details

The hydrogen atoms were placed at calculated positions with the SHELX program (AFIX options: 43 and 147).

Comment

The rational design of metal–organic complexes has received remarkable attention and has developed rapidly due to their fascinating potential applications in functional materials, [4–11]. All aspects of this research hotspot have been deeply discussed by some recent reviews, which show that the diverse structures of such materials are always dependent on many factors, such as substituent and number of coordination sites provided by organic ligands, ligand to metal salt ratio, template, metal ion, pH value, and counteranion [12]. In recent years, one of our interests focuses on the coordination assembly based on bis(pyridine) ligands and aromatic polycarboxylic acids ligands. In 4,4'-dipyridylsulfide ligand, two pyridine rings can keep different dihedral angles around the sulphur atom in the process of forming a complex. On the other hand, the aromatic polycarboxylic acids can form complexes with metal ions in flexible way, which can enhance stability of the complex [13]. As a continuation of our previous investigation, in this study we reacted 5-aminoisophthalic acid with Ni^{2+} ions, then employed 4,4'-dipyridylsulfide ligand as an auxiliary ligand, which features di-nuclear structure.

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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}
Ni1	0.57627(3)	0.10138(9)	0.63307(3)	0.0212(2)
S1	0.80599(7)	0.2223(3)	0.67242(11)	0.0652(6)
O1	0.57280(13)	0.4063(5)	0.58585(17)	0.0249(9)
O2	0.56661(13)	0.1019(5)	0.53238(16)	0.0256(9)
O3	0.56631(19)	0.0982(6)	0.3055(2)	0.0575(15)
O4	0.56615(16)	0.4049(5)	0.25372(18)	0.0378(11)
O5	0.58060(15)	−0.2178(5)	0.65261(17)	0.0334(11)
H5A	0.5627	−0.2885	0.6198	0.050*
H5B	0.5685	−0.2438	0.6830	0.050*
O6	0.58464(14)	0.1826(5)	0.72697(17)	0.0308(10)
H6A	0.5697	0.0925	0.7445	0.046*
H6B	0.5706	0.3046	0.7286	0.046*
N1	0.64940(17)	0.1213(7)	0.6505(2)	0.0286(11)
N2	0.8313(3)	−0.2014(14)	0.5131(4)	0.087(2)
N3	0.49946(15)	0.9199(6)	0.3904(2)	0.0233(10)
H3A	0.5057	0.9903	0.3583	0.028*
H3B	0.5106	0.9992	0.4262	0.028*
C1	0.55905(19)	0.4234(7)	0.4704(2)	0.0195(12)
C2	0.5677(2)	0.3199(7)	0.4182(3)	0.0218(12)
H2	0.5818	0.1846	0.4243	0.026*
C3	0.55574(19)	0.4151(7)	0.3580(3)	0.0214(12)
C4	0.53403(19)	0.6184(7)	0.3488(3)	0.0228(12)
H4	0.5254	0.6834	0.3080	0.027*
C5	0.52547(19)	0.7222(7)	0.4007(3)	0.0184(12)
C6	0.53922(19)	0.6278(7)	0.4617(3)	0.0232(13)
H6	0.5352	0.7014	0.4969	0.028*
C7	0.56724(19)	0.3064(8)	0.5330(2)	0.0193(12)
C8	0.5637(2)	0.2998(8)	0.3014(3)	0.0281(14)
C9	0.6754(2)	0.2795(10)	0.6852(3)	0.0412(17)
H9	0.6608	0.3733	0.7064	0.049*
C10	0.7227(2)	0.3138(10)	0.6919(3)	0.0454(18)
H10	0.7392	0.4281	0.7166	0.055*
C11	0.7452(2)	0.1738(9)	0.6610(3)	0.0376(15)
C12	0.7185(2)	0.0119(10)	0.6247(3)	0.0481(18)
H12	0.7321	−0.0827	0.6026	0.058*
C13	0.6716(2)	−0.0120(9)	0.6206(3)	0.0408(17)
H13	0.6544	−0.1251	0.5960	0.049*
C14	0.8157(2)	0.0551(11)	0.6104(4)	0.0466(18)
C15	0.8058(3)	0.1311(12)	0.5483(4)	0.059(2)
H15	0.7936	0.2687	0.5375	0.070*
C16	0.8143(3)	0.0006(16)	0.5025(4)	0.077(3)
H16	0.8077	0.0556	0.4607	0.093*
C17	0.8410(3)	−0.2613(12)	0.5739(5)	0.071(3)
H17	0.8541	−0.3975	0.5844	0.085*
C18	0.8339(3)	−0.1423(11)	0.6247(4)	0.062(2)
H18	0.8415	−0.1979	0.6667	0.074*

The asymmetric unit of title complex contains one independent Ni(II) cation, one AIP^{2−} anion, two coordinated H₂O and one dps ligand. Each Ni center is six coordinated by two carboxylato O atoms from one AIP^{2−} ligand, two N atoms from one dps ligand and one AIP^{2−} ligand and two water molecules to form a slight distorted octahedral geometry

(see the figure). The two carboxylic O atoms of the AIP^{2−} molecule are coordinated with Ni(II) by chelate mode. The Ni—O lengths are in the range of 2.029(4)–2.140(4) Å. The Ni—N lengths are 2.144(5) (Ni–N1A) and 2.080(6) (Ni–N2) Å. Two carboxylate groups of each AIP^{2−} ligand have a dihedral angle of 23.49° and 26.17° toward the plane of the corresponding linking phenyl rings, respectively. The dps ligand shows obvious torsion with a dihedral angle between the two pyridine rings of 80.39°.

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