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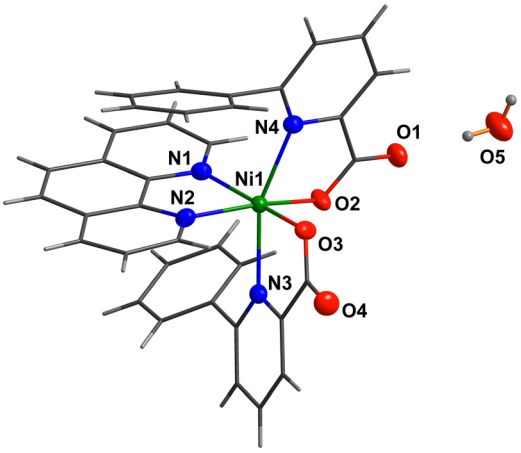
The crystal structure of [(1,10-phenanthroline-κ²*N*,*N*)-bis(6-phenylpyridine-2-carboxylate-κ²*N*,*O*) nickel(II)] monohydrate, $C_{36}H_{26}N_4O_5Ni$



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Abstract

$C_{36}H_{26}N_4O_5Ni$, orthorhombic, $P2_12_12_1$ (no. 19), a = 10.5860(9) Å, b = 10.6497(6) Å, c = 26.4492(15) Å, β = 90°, V = 2981.8(3) Å^3, Z = 4, $R_{gt}(F)$ = 0.0341, $wR_{ref}(F^2)$ = 0.0703, T = 200 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.

Table 1: Data collection and handling.

Crystal:	Grey block
Size:	0.13 × 0.10 × 0.08 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.70 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	13,389, 5193, 0.036
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 4857
$N(param)_{refined}$:	418
Programs:	Bruker [1], Olex2 [2], SHELX [3], Diamond [4]

Source of material

The title compound has been synthesized as following: 1,10-phenanthroline (0.0405 g, 0.25 mmol), 6-phenylpyridine-2-carboxylic acid (0.0498 g, 0.25 mmol), $Ni(O_2CMe)_2{\cdot}4H_2O$ (0.0622 g, 0.25 mmol), and NaOH (0.010 g, 0.25 mmol) were dissolved into a solution of 22 mL 95% ethanol-water (v:v = 1:1) under constant heating and stirring. After the mixture was heated up to 70 °C, the mixture continued to react for 5 h at this temperature. Then the reaction was then stopped and cooled down to room temperature and filtered. The needle-like crystals of [(1,10-phenanthroline-k²*N*,*N*)-bis(6-phenylpyridine-2-carboxylate-k²*N*,*O*) nickel(II)] monohydrate were received in 15 days. **Anal. Calcd.** for $C_{36}H_{26}N_4O_5Ni$: C, 66.12; H, 3.98; N, 8.57. Found: C, 65.96; H, 4.28; N, 8.39. **IR**: 1655 cm^{-1} (C=O), 1386 cm^{-1} (COO–).

Experimental details

The hydrogen atoms were positioned geometrically (C–H = 0.93 Å and O–H = 0.85 Å). Their U_{iso} values were set to 1.2U_{eq} or 1.5U_{eq} of the parent atoms.

Comment

In general, transition metal complexes may be self-assembled to create 1D, 2D, and 3D structures [5], and they have also exhibited potential applications in anti-tumor agents [6], antiviral agents [7], CO_2 photoreduction [8], effective adsorption of iodine [9], and so on. In our previous work, Co(II), Cu(II), Zn(II), and Pb(II) complexes

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Ni1	0.42090 (4)	0.45207 (4)	0.59368 (2)	0.01911 (13)
O1	0.3875 (2)	0.5324 (2)	0.44548 (8)	0.0300 (6)
O2	0.4389 (2)	0.4422 (2)	0.51826 (8)	0.0257 (6)
O3	0.5252 (2)	0.6081 (2)	0.60239 (10)	0.0256 (6)
O4	0.7116 (3)	0.6801 (3)	0.62811 (10)	0.0316 (7)
N1	0.2961 (3)	0.3006 (3)	0.59761 (11)	0.0209 (7)
N2	0.4120 (3)	0.4213 (3)	0.67260 (10)	0.0207 (7)
N3	0.6123 (3)	0.3701 (3)	0.59883 (11)	0.0194 (7)
N4	0.2747 (3)	0.5821 (3)	0.57025 (10)	0.0211 (7)
C1	0.1971 (3)	0.6577 (3)	0.59669 (15)	0.0254 (8)
C2	0.1260 (4)	0.7506 (4)	0.57310 (16)	0.0343 (11)
H2	0.072651	0.801209	0.592179	0.041*
C3	0.1343 (4)	0.7678 (4)	0.52176 (15)	0.0364 (11)
H3	0.086414	0.829247	0.505754	0.044*
C4	0.2154 (4)	0.6921 (4)	0.49427 (14)	0.0300 (10)
H4	0.223806	0.702079	0.459503	0.036*
C5	0.2834 (3)	0.6013 (3)	0.51969 (13)	0.0207 (8)
C6	0.3763 (3)	0.5195 (3)	0.49157 (13)	0.0208 (8)
C7	0.1873 (4)	0.6400 (4)	0.65239 (14)	0.0284 (9)
C8	0.1353 (4)	0.5331 (4)	0.67279 (15)	0.0323 (9)
H8	0.107874	0.469324	0.651406	0.039*
C9	0.1230 (4)	0.5184 (5)	0.72486 (17)	0.0451 (13)
H9	0.087862	0.445627	0.738262	0.054*
C10	0.1636 (5)	0.6134 (5)	0.75619 (18)	0.0581 (15)
H10	0.155221	0.605219	0.791035	0.070*
C11	0.2163 (6)	0.7202 (5)	0.73622 (18)	0.0629 (16)
H11	0.244869	0.783122	0.757743	0.075*
C12	0.2274 (5)	0.7353 (4)	0.68453 (17)	0.0459 (12)
H12	0.261494	0.808736	0.671309	0.055*
C13	0.6403 (4)	0.5954 (4)	0.61431 (13)	0.0218 (9)
C14	0.6930 (3)	0.4627 (4)	0.61195 (12)	0.0210 (8)
C15	0.8180 (4)	0.4432 (4)	0.62362 (15)	0.0350 (10)
H15	0.869763	0.509751	0.633052	0.042*
C16	0.8648 (4)	0.3223 (4)	0.62098 (18)	0.0460 (13)
H16	0.948928	0.305788	0.628569	0.055*
C17	0.7854 (4)	0.2274 (4)	0.60705 (15)	0.0359 (11)
H17	0.815813	0.145621	0.604867	0.043*
C18	0.6593 (3)	0.2521 (3)	0.59611 (14)	0.0228 (8)
C19	0.5761 (4)	0.1462 (3)	0.57991 (12)	0.0225 (8)
C20	0.5784 (4)	0.0352 (4)	0.60725 (13)	0.0328 (9)
H20	0.627489	0.029757	0.636336	0.039*
C21	0.5091 (4)	−0.0669 (4)	0.59191 (18)	0.0400 (10)
H21	0.509387	−0.140261	0.611006	0.048*
C22	0.4390 (4)	−0.0602 (4)	0.54806 (17)	0.0419 (11)
H22	0.392266	−0.129349	0.537443	0.050*
C23	0.4380 (4)	0.0488 (4)	0.51992 (16)	0.0397 (10)
H23	0.391286	0.052742	0.490179	0.048*
C24	0.5062 (4)	0.1521 (4)	0.53580 (14)	0.0285 (9)
H24	0.505015	0.225610	0.516834	0.034*
C25	0.2302 (4)	0.2488 (4)	0.56053 (14)	0.0291 (9)
H25	0.239324	0.281038	0.528052	0.035*
C26	0.1474 (4)	0.1478 (4)	0.56792 (16)	0.0332 (10)
H26	0.101635	0.115130	0.540934	0.040*
C27	0.1347 (4)	0.0980 (4)	0.61500 (16)	0.0324 (10)
H27	0.081213	0.029989	0.620394	0.039*
C28	0.2032 (3)	0.1502 (4)	0.65550 (15)	0.0265 (9)
C29	0.2814 (3)	0.2527 (3)	0.64467 (13)	0.0202 (8)
C30	0.3468 (3)	0.3152 (3)	0.68517 (13)	0.0206 (8)
C31	0.3368 (4)	0.2702 (4)	0.73465 (14)	0.0267 (9)
C32	0.2571 (4)	0.1638 (4)	0.74430 (15)	0.0320 (10)
H32	0.249857	0.133421	0.777126	0.038*
C33	0.1928 (4)	0.1072 (4)	0.70680 (15)	0.0333 (10)
H33	0.140814	0.039246	0.714218	0.040*
C34	0.4686 (4)	0.4822 (4)	0.70948 (13)	0.0289 (10)
H34	0.511335	0.556034	0.701673	0.035*
C35	0.4679 (4)	0.4418 (4)	0.75993 (13)	0.0343 (10)
H35	0.511949	0.486319	0.784505	0.041*
C36	0.4020 (4)	0.3363 (4)	0.77255 (13)	0.0320 (10)
H36	0.400211	0.308367	0.805858	0.038*
O5	0.3829 (3)	0.6157 (3)	0.34645 (10)	0.0465 (9)
H5A	0.337911	0.681791	0.345563	0.070*
H5B	0.385693	0.587944	0.376572	0.070*

of 6-phenylpyridine-2-carboxylates have been synthesized and structurally characterized [10–13].

The title compound was characterized through elemental analysis, infrared spectrum, and X-ray single-crystal diffraction. The molecular structure is composed of a Ni(II) ion, two 6-phenylpyridine-2-carboxylate ligands, one 1,10-phenanthroline ligand and one lattice water molecule. The Ni(II) ion coordinates with four N atoms (N1, N2, N3, N4). N1 and N2 atoms are from one 1,10-phenanthroline ligand, and N3, N4 atoms are from two different 6-phenylpyridine-2-carboxylate ligands, and two O atoms (O2, O3) from two different 6-phenylpyridine-2-carboxylate ligands, forming a distorted six-coordinated octahedral geometry. The Ni–O and Ni–N distances are 2.007(2) Å (Ni1–O2), 2.008(3) Å (Ni1–O3), 2.087(3) Å (Ni1–N1), 2.115(3) Å (Ni1–N2), 2.211(3) Å (Ni1–N3), 2.167(3) Å (Ni1–N4), which is consistent with the literature. The sum of angles around Ni(II) ion is 362.59° (N3–Ni1–O2, 87.34(10)°; N3–Ni1–N2, 85.36(11)°; N2–Ni1–N4, 110.44(11)°; N4–Ni1–O2, 79.45(10)°), and the angle of O3–Ni1–N1 is 168.97(11)°, showing that O2, N3, N2, N4 atoms are at the equatorial plane and O3, N1 atoms are at axial position. The dihedral angles between phenyl and pyridyl groups of the 6-phenylpyridine-2-carboxylate ligands are 66.0° (phenyl ring: C7–C8–C9–C10–C11–C12 and pyridyl ring: C1–C2–C3–C4–C5–N4) and 130.3° (phenyl ring: C19–C20–C21–C22–C23–C24 and pyridyl ring: C14–C15–C16–C17–C18–N3). The molecules form 1D chain structure by the O–H···O hydrogen bond between the lattice water molecule and a 6-phenylpyridine-2-carboxylate ligand.

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

References

1. Bruker. SAINT and SADABS; Bruker AXS Inc.: Madison, Wisconsin, USA, 2000.
2. 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.
3. Sheldrick G. M. Crystal structure refinement with SHELXL. *Acta Crystallogr.* 2015, *C71*, 3–8.
4. Brandenburg K. DIAMOND. Visual Crystal Structure Information System (ver. 3.2); Crystal Impact: Bonn, Germany, 2012.
5. Huang Y. J., Zhang J. Synthesis and characterization a series of norfloxacin-transition metal complexes. *Inorg. Chim. Acta* 2020, *501*, 119244.
6. Renata P., Małgorzata W. S., Sabina J., Tomasz K., Marcelina K., Anna H. B. Latest developments in metal complexes as anticancer agents. *Coord. Chem. Rev.* 2022, *452*, 214307.
7. Johannes K., Seth M. C. Metal complexes as antiviral agents for SARS-CoV-2. *ChemBioChem* 2021, *22*, 2600–2607.
8. Qi X. J., Zhong R. L., Chen M. M., Sun C. Y., You S. Q., Gu J. X., Shan G. G., Cui D. X., Wang X. L., Su Z. M. Single metal-organic cage decorated with an Ir(III) complex for CO_2 photoreduction. *ACS Catal.* 2021, *11*, 7241–7248.
9. Qin J. X., Zhang W., Chen Y. T., Liu R., Fan Y. R. Zinc-based triazole metal complexes for efficient iodine adsorption in water. *Environ. Sci. Pollut. Res. Int.* 2021, *28*, 28797–28807.
10. Tai X.-S., Wang Z.-J., Ouyang J., Li Y.-F., Zhang W., Jia W.-L., Wang L.-H. The crystal structure of [(phenantroline-κ^2N,N')-bis(6-phenylpyridine-2-carboxylate-κ^2N,O)cobalt(II)] monohydrate, $C_{36}H_{26}N_4O_5Co$. *Z. Kristallogr. N. Cryst. Struct.* 2021, *236*, 1309–1311.
11. Wang L.-H., Liang L., Li X.-T., Cao S.-H., Tai X.-S. The crystal structure of bis(6-phenylpyridine-2-carboxylato-κ^2N,O)copper(II), $C_{24}H_{16}N_2O_4Cu$. *Z. Kristallogr. N. Cryst. Struct.* 2021, *236*, 1251–1253.
12. Wang L.-H., Wang Z.-J., Ouyang J., Tai X.-S. The crystal structure of bis(6-phenylpyridine-2-carboxylate-κ^2N,O)-(2,2′-bipyridine-κ^2N,N') zinc(II) monohydrate, $C_{34}H_{26}N_4O_5Zn$. *Z. Kristallogr. N. Cryst. Struct.* 2021, *236*, 1297–1299.
13. Tai X. S., Liang L., Li X. T., Cao S. H., Wang L. H. Crystal structure of diaqua-bis(μ_2-6-phenylpyridine-2-carboxylate-$\kappa^3N,O:O$)-bis(6-phenylpyridine-2-carboxylato-κ^2N,O)lead(II) – *N,N*-dimethylformamide – water (1/2/4), $C_{54}H_{58}N_6O_{16}Pb_2$. *Z. Kristallogr. N. Cryst. Struct.* 2021, *236*, 1199–1201.