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Crystal structure of [(bis(1,10-phenanthroline- κ^2N,N')-(2-carboxy-4-(3-carboxy-5-carboxylatophenoxy)benzoato- $\kappa^2O:O'$))nickel(II) monohydrate, (1,10-phenanthroline- $\kappa^2N:N'$)-(μ_2 -(5-(3',4'-dicarboxylphenoxy)-isophthalate- $\kappa^2O:O'$))nickel(II)], $C_{40}H_{24}N_4O_9Ni \cdot H_2O$

Table 1: Data collection and handling.

Crystal:	Green block
Size:	0.42 × 0.38 × 0.29 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.64 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω
θ_{max} , completeness:	25.5°, 98%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	15923, 5769, 0.041
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 4660
$N(param)_{refined}$:	489
Programs:	CrysAlis ^{PRO} [1], SHELX [2, 3]

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

Atom	x	y	z	U_{iso}^*/U_{eq}
Ni1	0.41847(4)	0.20379(4)	0.72740(3)	0.03181(15)
O1	0.27560(19)	0.35556(19)	0.72450(15)	0.0340(5)
O2	0.3639(2)	0.3007(2)	0.85872(16)	0.0405(6)
O3	0.2151(3)	0.5396(3)	1.1621(2)	0.0746(10)
O4	0.0507(3)	0.6835(3)	1.15885(19)	0.0648(9)
H4	0.0557	0.6851	1.2188	0.097*
O5	0.0171(3)	0.6891(3)	0.34996(19)	0.0648(8)
O6	0.0937(3)	0.8486(3)	0.3318(2)	0.0732(9)
O7	0.1794(3)	0.9460(3)	0.4350(2)	0.0737(9)
H7	0.1414	0.9200	0.4016	0.110*
O8	0.2022(3)	0.9317(3)	0.5940(3)	0.0895(11)
O9	−0.06605(19)	0.7086(2)	0.82317(16)	0.0420(6)
C1	0.0453(3)	0.7693(4)	0.3859(3)	0.0498(11)
C2	0.0183(3)	0.7646(3)	0.5029(2)	0.0369(8)
C3	0.0684(3)	0.8219(3)	0.5638(2)	0.0364(8)
C4	0.0375(3)	0.7998(3)	0.6704(2)	0.0364(8)
H4A	0.0711	0.8353	0.7118	0.044*
C5	−0.0399(3)	0.7279(3)	0.7153(2)	0.0341(8)
C6	−0.0930(3)	0.6748(4)	0.6580(3)	0.0470(10)
H6	−0.1480	0.6278	0.6889	0.056*
C7	−0.0617(3)	0.6936(4)	0.5518(3)	0.0498(10)
H7A	−0.0960	0.6569	0.5118	0.060*
C8	0.0278(3)	0.6347(3)	0.8668(2)	0.0284(7)
C9	0.0347(3)	0.6548(3)	0.9641(2)	0.0318(8)
H9	−0.0212	0.7158	0.9970	0.038*
C10	0.1263(3)	0.5828(3)	1.0131(2)	0.0308(7)
C11	0.2087(3)	0.4917(3)	0.9638(2)	0.0323(8)
H11	0.2703	0.4443	0.9959	0.039*

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Abstract

$C_{40}H_{26}N_4O_{10}Ni$, triclinic, $P\bar{1}$ (no. 2), $a = 11.5851(8)$ Å, $b = 11.5875(7)$ Å, $c = 13.3921(8)$ Å, $\alpha = 81.440(5)^\circ$, $\beta = 77.913(6)^\circ$, $\gamma = 76.014(6)^\circ$, $V = 1696.65(18)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0516$, $wR_{ref}(F^2) = 0.1203$, $T = 293(2)$ K.

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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

A mixture of $Ni(OAc)_2 \cdot 4H_2O$ (0.1 mmol, 25 mg), 1,10-phenanthroline (0.1 mmol, 18 mg) and 5-(3',4'-dicarboxylphenoxy)-isophthalic acid (H4dic) (50 mg,

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Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
C12	0.1994(3)	0.4709(3)	0.8666(2)	0.0285(7)
C13	0.1080(3)	0.5430(3)	0.8184(2)	0.0290(7)
H13	0.1012	0.5292	0.7535	0.035*
C14	0.1561(4)	0.9040(4)	0.5306(3)	0.0527(10)
C15	0.1358(3)	0.5997(3)	1.1193(3)	0.0390(9)
C16	0.2844(3)	0.3706(3)	0.8134(2)	0.0306(7)
C17	0.3700(3)	0.1145(3)	0.5385(3)	0.0469(10)
H17	0.3236	0.0670	0.5836	0.056*
C18	0.3724(4)	0.1191(4)	0.4334(3)	0.0586(12)
H18	0.3277	0.0762	0.4092	0.070*
C19	0.4410(4)	0.1870(4)	0.3671(3)	0.0598(12)
H19	0.4427	0.1911	0.2969	0.072*
C20	0.5094(3)	0.2511(3)	0.4035(3)	0.0459(9)
C21	0.4994(3)	0.2432(3)	0.5100(2)	0.0333(8)
C22	0.5843(4)	0.3242(4)	0.3400(3)	0.0618(12)
H22	0.5919	0.3293	0.2691	0.074*
C23	0.6443(4)	0.3859(4)	0.3804(3)	0.0576(11)
H23	0.6932	0.4322	0.3369	0.069*
C24	0.6348(3)	0.3821(3)	0.4882(3)	0.0422(9)
C25	0.5624(3)	0.3108(3)	0.5533(2)	0.0321(8)
C26	0.6945(3)	0.4451(4)	0.5351(4)	0.0555(11)
H26	0.7439	0.4934	0.4950	0.067*
C27	0.6801(4)	0.4354(4)	0.6385(4)	0.0583(11)
H27	0.7205	0.4760	0.6700	0.070*
C28	0.6046(3)	0.3644(3)	0.6976(3)	0.0479(10)
H28	0.5946	0.3598	0.7687	0.057*
C29	0.1838(3)	0.1112(4)	0.7872(3)	0.0460(9)
H29	0.1490	0.1780	0.7472	0.055*
C30	0.1111(4)	0.0333(4)	0.8401(3)	0.0569(11)
H30	0.0301	0.0476	0.8343	0.068*
C31	0.1606(4)	−0.0639(4)	0.9002(3)	0.0615(12)
H31	0.1130	−0.1156	0.9369	0.074*
C32	0.2841(3)	−0.0858(3)	0.9065(3)	0.0474(10)
C33	0.3496(3)	−0.0041(3)	0.8498(2)	0.0358(8)
C34	0.3456(4)	−0.1853(4)	0.9660(3)	0.0653(12)
H34	0.3029	−0.2408	1.0035	0.078*
C35	0.4633(4)	−0.2006(4)	0.9689(3)	0.0598(11)
H35	0.5002	−0.2657	1.0094	0.072*
C36	0.5333(3)	−0.1188(3)	0.9112(3)	0.0431(9)
C37	0.4758(3)	−0.0203(3)	0.8515(2)	0.0344(8)
C38	0.6579(4)	−0.1305(4)	0.9086(3)	0.0544(11)
H38	0.7001	−0.1941	0.9469	0.065*
C39	0.7154(3)	−0.0486(4)	0.8500(3)	0.0536(11)
H39	0.7977	−0.0561	0.8474	0.064*
C40	0.6520(3)	0.0464(3)	0.7939(3)	0.0443(9)
H40	0.6932	0.1023	0.7548	0.053*
N1	0.5457(2)	0.3027(2)	0.6571(2)	0.0351(7)
N2	0.4312(2)	0.1752(2)	0.5768(2)	0.0334(7)
N3	0.5350(2)	0.0614(2)	0.7934(2)	0.0353(7)
N4	0.2990(2)	0.0942(2)	0.7916(2)	0.0341(7)

0.1 mmol), was dissolved in 8 mL H₂O. Then the solution was heated in a 23 mL Teflon-lined autoclave under autogenous pressure at 393 K for three days. After cooling to room temperature green crystals formed.

Experimental details

Their U_{iso} values were set to $1.2U_{eq}$ of the parent atoms. The water molecule was omitted.

Comment

Mixed ligands-MOFs can be constructed from metal ions and carboxylic acid and nitrogen-containing ligands. In most situations, N-containing ligands are electrically neutral, while carboxylate ligands are negatively charged. Among organic aromatic multicarboxylates like the ether oxygen tetracarboxylates can yield networks and have been widely utilized to construct MOFs [4–6]. On the other hand, 1,10-phenanthroline serves as bridging ligand to coordinate to two metal ions [7].

In the title complex, there is one Ni(II) ion, one dianionic H₂dic ligand, and two 1,10-phenanthroline ligands in the asymmetric unit. Ni1 is six-coordinated with a distorted octahedral geometry by two O atoms from one H₂dic, and four N atoms from the 1,10-phenanthroline ligands from the axial positions (see the figure). Thus a mononuclear complex is present [8]. The Ni–O bond lengths are 2.106(2) Å; 2.129(2) Å and the Ni–N ones are in the range of 2.061(3) to 2.077(3) Å. The bond angles of N–Ni–N are in the range of 80.21(11)° to 175.66(10)°.

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