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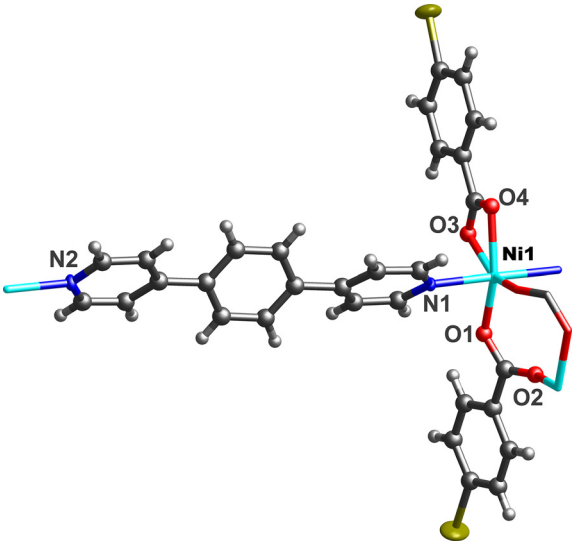
Crystal structure of *catena*-poly[(μ_2-1,4-di(pyridin-4-yl)benzene-κ^2N:N')-(4-bromobenzoate-κ^2O:O')-(μ-2-bromobenzoate-κ^2O,O')nickel(II)] – water (2/1), $C_{30}H_{21}Br_2N_2NiO_{4.5}$



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Abstract

$C_{30}H_{21}Br_2N_2NiO_{4.5}$, triclinic, $P\bar{1}$ (no. 2), a = 8.7895(17) Å, b = 13.167(3) Å, c = 14.067(3) Å, α = 109.049(3)°, β = 92.619(3)°, γ = 108.533(3)°, V = 1438.6(5) Å^3, Z = 2, $R_{gt}(F)$ = 0.0496, $wR_{ref}(F^2)$ = 0.1003, T = 296(2) K.

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A part of the title crystal structure is shown in the figure (the solvent water molecule and the disordered Br atom were omitted in the figure for clarity). 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 1: Data collection and handling.

Crystal:	Green block
Size:	0.20 × 0.18 × 0.15 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	3.49 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	25.0°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7392, 5043, 0.035
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3172
$N(param)_{refined}$:	371
Programs:	Bruker [1], SHELX [2]

1 Source of material

A mixture of $Ni(NO_3)_2{\cdot}6H_2O$ (1 mmol, 290.8 mg), 1,4-di(pyridin-4-yl)benzene (dpb) (1 mmol, 232.3 mg), 4-bromobenzoic acid (Hbba) (2 mmol, 402.0 mg), *N*, *N*-dimethylacetamide (1 mL) and H_2O (3 mL) were sealed in a 25 mL vial and heated at 393 K for three days. Green block crystals were collected.

2 Experimental details

The C-bound H atoms were placed in idealized positions (C–H = 0.93 Å) and refined by the riding model with U_{iso}(H) = 1.2 U_{eq}(C). The O-bound H atoms were refined with restrained bond lengths (O–H = 0.82 Å). The U_{iso}(H) = 1.5 U_{eq}(O). One Br atom is disordered over two positions. The two parts overlapped each other with site occupation factors of 0.65 and 0.35.

3 Comment

As a halogenobenzoic acid, 4-bromobenzoic acid was widely used as organic ligand to construct various metal coordination polymers [3–9]. However, the Ni(II) coordination polymers based on 4-bromobenzoic acid were rarely reported. From the Cambridge Structural Database (Version 5.44, April 2023), only three structures of Ni(II) coordination polymers were found [10–12]. To further explore this research area, we

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

Atom	x	y	z	U_{iso}*/U_{eq}
Br1	0.62597 (11)	0.50233 (7)	0.69669 (6)	0.1071 (3)
Br2[a]	1.3167 (6)	1.2065 (5)	−0.0047 (4)	0.0800 (11)
Br2B[b]	1.3320 (18)	1.181 (2)	−0.0107 (8)	0.112 (3)
Ni1	0.66499 (7)	0.99945 (5)	0.39093 (4)	0.02806 (17)
O1	0.6600 (3)	0.8845 (2)	0.4581 (2)	0.0353 (7)
O2	0.5307 (3)	0.9480 (2)	0.5850 (2)	0.0370 (7)
O3	0.8699 (3)	0.9865 (2)	0.3180 (2)	0.0366 (8)
O4	0.7440 (4)	1.0977 (3)	0.2946 (2)	0.0403 (8)
N1	0.5128 (4)	0.8612 (3)	0.2650 (2)	0.0296 (8)
N2	−0.1841 (4)	0.1361 (3)	−0.4819 (2)	0.0340 (9)
C1	0.5972 (5)	0.8811 (4)	0.5368 (3)	0.0306 (10)
C2	0.6081 (5)	0.7894 (4)	0.5761 (3)	0.0320 (11)
C3	0.6993 (7)	0.7234 (4)	0.5360 (4)	0.0592 (15)
H3	0.756521	0.735655	0.484399	0.071*
C4	0.7066 (8)	0.6379 (5)	0.5721 (5)	0.0804 (19)
H4	0.769809	0.593907	0.545722	0.096*
C5	0.6188 (7)	0.6197 (5)	0.6474 (4)	0.0641 (16)
C6	0.5264 (6)	0.6833 (4)	0.6872 (4)	0.0551 (14)
H6	0.467107	0.669824	0.737669	0.066*
C7	0.5222 (6)	0.7680 (4)	0.6515 (3)	0.0437 (12)
H7	0.459730	0.812201	0.678919	0.052*
C8	0.8556 (5)	1.0561 (4)	0.2788 (3)	0.0340 (11)
C9	0.9698 (5)	1.0897 (4)	0.2084 (3)	0.0381 (12)
C10	1.0818 (6)	1.0360 (4)	0.1787 (4)	0.0536 (14)
H10	1.087833	0.979154	0.202716	0.064*
C11	1.1858 (6)	1.0677 (5)	0.1125 (4)	0.0632 (16)
H11	1.260133	1.031541	0.091387	0.076*
C12	1.1758 (6)	1.1533 (5)	0.0793 (4)	0.0559 (15)
C13	1.0684 (7)	1.2072 (5)	0.1095 (4)	0.0727 (18)
H13	1.064184	1.265173	0.086688	0.087*
C14	0.9654 (7)	1.1757 (5)	0.1743 (4)	0.0617 (16)
H14	0.892292	1.213002	0.195225	0.074*
C15	0.3997 (5)	0.8716 (4)	0.2055 (3)	0.0403 (12)
H15	0.381043	0.940653	0.225401	0.048*
C16	0.3093 (6)	0.7854 (4)	0.1162 (3)	0.0443 (13)
H16	0.233699	0.797591	0.076935	0.053*
C17	0.3315 (5)	0.6796 (4)	0.0848 (3)	0.0328 (11)
C18	0.4456 (5)	0.6683 (4)	0.1477 (3)	0.0336 (11)
H18	0.464696	0.599522	0.130885	0.040*
C19	0.5316 (5)	0.7597 (3)	0.2359 (3)	0.0327 (11)
H19	0.606759	0.749497	0.277199	0.039*
C20	0.2453 (5)	0.5857 (4)	−0.0141 (3)	0.0348 (11)
C21	0.2066 (6)	0.6111 (4)	−0.0983 (3)	0.0506 (14)
H21	0.234102	0.687367	−0.092786	0.061*
C22	0.1276 (6)	0.5243 (4)	−0.1906 (3)	0.0507 (14)
H22	0.103719	0.543022	−0.246348	0.061*
C23	0.0834 (5)	0.4098 (4)	−0.2011 (3)	0.0360 (11)
C24	0.1225 (6)	0.3853 (4)	−0.1170 (3)	0.0442 (13)
H24	0.095027	0.309123	−0.122518	0.053*
C25	0.2016 (6)	0.4716 (4)	−0.0246 (3)	0.0437 (13)
H25	0.225625	0.452561	0.030987	0.052*
C26	−0.0048 (5)	0.3159 (4)	−0.3004 (3)	0.0340 (11)
C27	−0.1010 (6)	0.3328 (4)	−0.3706 (4)	0.0533 (15)
H27	−0.107315	0.405048	−0.358468	0.064*
C28	−0.1878 (6)	0.2414 (4)	−0.4592 (3)	0.0499 (14)
H28	−0.251808	0.254715	−0.505036	0.060*
C29	−0.0862 (5)	0.1205 (4)	−0.4162 (3)	0.0348 (11)
H29	−0.077952	0.048372	−0.431793	0.042*
C30	0.0027 (5)	0.2067 (4)	−0.3265 (3)	0.0367 (11)
H30	0.068199	0.191472	−0.283113	0.044*
O1W[c]	0.5906 (16)	0.0915 (12)	0.0938 (11)	0.202 (6)
H1A[c]	0.623260	0.083495	0.145620	0.302*
H1B[c]	0.511424	0.112166	0.096330	0.302*

[a]Occupancy: 0.65 (3), [b]Occupancy: 0.35 (3), [c]Occupancy: 0.5.

chose 4-bromobenzoic acid (bbaH) as the organic ligand and the rigid N-containing ligand of 1,4-di(pyridin-4-yl)benzene (dpb) as the auxiliary bridging ligand [13] to generate a new Ni(II) coordination polymer.

The asymmetric unit consists of one Ni(II) cation, one dpb ligand, two bba^- ligands and one half of a free water solvent. The bond distances of Ni–O/N (2.020(3)–2.157(3) Å) are all in normal ranges and the bond angles around Ni(II) cation vary from 61.27(1)° to 179.18(14)°. The Ni(II) cation is six coordinated by four O atoms from three bba^- ligands and two N atoms from two symmetry related dpb ligands to furnish a distorted octahedral coordination geometry. The deprotonated bba^- ligands adopt a bidentate chelate mode and a bidentate bridging mode, to link the neighboring Ni(II) cations forming a $Ni_2(COO)_4$ subunit (see the figure).

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