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Crystal structure of *catena*-poly[triaqua-(μ_2 -1,3-di(1*H*-imidazol-1-yl)propane- κ^2 N:N')-(4,4'-(1*H*-1,2,4-triazole-3,5-diyl)dibenzoato- κ^1 O)nickel(II)] *N,N'*-dimethylformamide (1/1), C₂₈H₃₅N₈O₈Ni

Figure 1: The asymmetric units of title coordination polymer.

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Abstract

C₂₈H₃₅N₈O₈Ni, monoclinic, *P*₂/c (no. 14), *a* = 14.7476(15) Å, *b* = 19.6335(19) Å, *c* = 10.8116(11) Å, β = 104.334(2)°, *V* = 3033.0(5) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0548, *wR*_{ref}(*F*²) = 0.0961, *T* = 293(2) K.

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A part of the title polymeric 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

The mixture of 3,5-bis(4'-carboxy-phenyl)-1,2,4-triazole 30.9 mg (0.1 mmol), nickel nitrate hexahydrate 29.1 mg

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.26 × 0.22 × 0.17 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.70 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	25.3°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	15,769, 5522, 0.073
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ (<i>I</i> _{obs}), 3303
<i>N</i> (<i>param</i>) _{refined} :	432
Programs:	Bruker [1], SHELX [2, 3]

(0.1 mmol), 1,3-di(1*H*-imidazol-1-yl)propane 20.1 mg (0.10 mmol), NaOH 6 mg (0.15 mmol) and *N,N*-dimethylformamide (6 mL) were placed in the autoclave lined with PTFE and heated at 120 °C for 24 h, then cooled to room temperature over 24 h. The green crystals after cooling were collected.

Experimental details

The structure was solved by direct methods and refined with the SHELX crystallographic software package [2]. The hydrogen atoms were placed at calculated positions and refined as riding atoms with isotropic displacement parameters [3].

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
C1	0.5129 (3)	0.1820 (2)	0.1597 (4)	0.0310 (10)
C2	0.4269 (2)	0.18077 (19)	0.2125 (3)	0.0257 (9)
C3	0.4347 (2)	0.1660 (2)	0.3407 (3)	0.0336 (10)
H3	0.493012	0.156058	0.394213	0.040*
C4	0.3560 (2)	0.1660 (2)	0.3887 (3)	0.0306 (9)
H4	0.361948	0.156183	0.474524	0.037*
C5	0.2683 (2)	0.18036 (18)	0.3106 (3)	0.0227 (9)
C6	0.2611 (2)	0.19732 (19)	0.1834 (3)	0.0279 (10)
H6	0.203052	0.208242	0.130288	0.034*
C7	0.3399 (3)	0.19795 (19)	0.1361 (3)	0.0288 (10)
H7	0.334493	0.210121	0.051411	0.035*
C8	0.1855 (2)	0.17554 (19)	0.3628 (3)	0.0227 (9)
C9	0.0948 (2)	0.16717 (19)	0.4848 (3)	0.0228 (8)
C10	0.0614 (2)	0.1638 (2)	0.6024 (3)	0.0251 (9)
C11	0.1252 (3)	0.1534 (2)	0.7181 (3)	0.0364 (11)
H11	0.188119	0.147297	0.720390	0.044*
C12	0.0966 (3)	0.1520 (2)	0.8300 (3)	0.0369 (11)
H12	0.140149	0.144604	0.907225	0.044*
C13	0.0030 (2)	0.16146 (19)	0.8281 (3)	0.0238 (9)
C14	−0.0608 (2)	0.17096 (19)	0.7125 (3)	0.0273 (9)
H14	−0.123706	0.176930	0.710248	0.033*
C15	−0.0323 (2)	0.1717 (2)	0.5996 (3)	0.0271 (9)
H15	−0.076123	0.177625	0.522106	0.032*
C16	−0.0253 (3)	0.1627 (2)	0.9535 (3)	0.0276 (9)
C17	0.7684 (4)	0.0651 (2)	0.0239 (4)	0.0527 (14)
H17	0.828604	0.082993	0.040842	0.063*
C18	0.6285 (3)	0.0495 (3)	0.0249 (5)	0.0573 (14)
H18	0.570206	0.054084	0.042424	0.069*
C19	0.6497 (4)	0.0028 (3)	−0.0561 (5)	0.0667 (15)
H19	0.609773	−0.029986	−0.102362	0.080*
C20	0.7984 (4)	−0.0256 (2)	−0.1226 (4)	0.0713 (18)
H20A	0.766382	−0.067714	−0.153503	0.086*
H20B	0.856383	−0.037408	−0.061700	0.086*
C21	0.8221 (4)	0.0115 (3)	−0.2357 (4)	0.0756 (18)
H21A	0.846710	0.056213	−0.207486	0.091*
H21B	0.871143	−0.013590	−0.261312	0.091*
C22	0.7445 (4)	0.0195 (3)	−0.3448 (4)	0.0703 (16)
H22A	0.700299	0.051208	−0.323584	0.084*
H22B	0.713110	−0.024069	−0.364077	0.084*
C23	0.7340 (3)	0.0971 (2)	−0.5340 (4)	0.0407 (11)
H23	0.690783	0.126965	−0.514044	0.049*
C24	0.8312 (4)	0.0134 (2)	−0.5200 (4)	0.0611 (15)
H24	0.868338	−0.024577	−0.492097	0.073*
C25	0.8253 (4)	0.0493 (2)	−0.6280 (4)	0.0573 (14)
H25	0.858564	0.038966	−0.688352	0.069*
C26	0.4676 (4)	0.0434 (3)	0.6484 (6)	0.091 (2)
H26A	0.406804	0.036451	0.592247	0.136*
H26B	0.484162	0.004597	0.703381	0.136*
H26C	0.512848	0.049131	0.598844	0.136*
C27	0.3834 (4)	0.1175 (3)	0.7700 (5)	0.093 (2)
H27A	0.393532	0.157636	0.822223	0.139*
H27B	0.371103	0.079520	0.819444	0.139*
H27C	0.330833	0.124452	0.698446	0.139*
C28	0.5371 (4)	0.1458 (3)	0.7515 (5)	0.0632 (15)
H28	0.531669	0.183433	0.801543	0.076*

Table 2: (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
N1	0.18690 (19)	0.17251 (16)	0.4864 (3)	0.0263 (7)
N2	0.0978 (2)	0.17132 (16)	0.2922 (3)	0.0296 (8)
H2	0.081690	0.171886	0.210103	0.036*
N3	0.0372 (2)	0.16598 (18)	0.3688 (3)	0.0306 (8)
N4	0.7035 (2)	0.08836 (17)	0.0762 (3)	0.0352 (9)
N5	0.7399 (3)	0.01331 (18)	−0.0561 (3)	0.0461 (10)
N6	0.7712 (3)	0.04439 (19)	−0.4605 (3)	0.0476 (10)
N7	0.7652 (2)	0.10212 (17)	−0.6378 (3)	0.0337 (9)
N8	0.4661 (3)	0.1035 (2)	0.7245 (4)	0.0546 (11)
Ni2	0.72081 (3)	0.16722 (3)	0.20828 (4)	0.02630 (15)
O1	0.58827 (17)	0.16243 (14)	0.2340 (2)	0.0356 (7)
O2	0.50257 (18)	0.20317 (17)	0.0484 (3)	0.0559 (9)
O3	−0.10554 (17)	0.18374 (14)	0.9536 (2)	0.0384 (8)
O4	0.0334 (2)	0.14215 (17)	1.0490 (2)	0.0548 (9)
O5	0.6092 (3)	0.14041 (18)	0.7172 (4)	0.0832 (13)
O1W	0.85681 (17)	0.17914 (16)	0.1935 (3)	0.0356 (7)
O2W	0.67705 (19)	0.23989 (15)	0.0612 (2)	0.0355 (7)
O3W	0.7364 (2)	0.25457 (15)	0.3299 (3)	0.0347 (7)
H1WB	0.9052 (10)	0.177 (2)	0.2554 (15)	0.061 (16)*
H1WA	0.8774 (16)	0.188 (2)	0.1283 (14)	0.11 (2)*
H2WA	0.6960 (13)	0.2360 (18)	−0.0066 (12)	0.037 (12)*
H2WB	0.6193 (6)	0.2295 (18)	0.040 (2)	0.046 (14)*
H3WB	0.7023 (16)	0.2863 (11)	0.290 (3)	0.10 (2)*
H3WA	0.7894 (10)	0.2729 (13)	0.361 (4)	0.09 (2)*

Comment

The combination of dicarboxylate anions with N-donor auxiliary ligands is a good choice for the construction of novel topologies and networks. Compared to well-known N-donor ligands, the 1,3-di(1*H*-imidazol-1-yl)propane (diim) ligand [4–6], bearing alkyl spacers, is a simple bidentate organic building block, which has specific advantages of being flexible and at the same time not being overly lengthy. On the other hand, multicarboxylic acids like the 3,5-bis(4'-carboxy-phenyl)-1,2,4-triazole have been widely utilized to construct such networks [7–10]. Meanwhile, many complexes with similar structure had been reported [11, 12].

The title structure is a one-dimensional framework, which *via* hydrogen bonds forms three-dimensional supramolecules. As shown in Figure 1, the asymmetric unit of the title structure contains one Ni(II) cation, one free *N,N*-dimethylformamide, one 1,3-di(1*H*-imidazol-1-yl)propane, one dianionic 4,4'-(1*H*-1,2,4-triazole-3,5-diyl)dibenzoate (bct) and three coordinated water molecules. Each Ni(II) is six-coordinated by three oxygen atoms from three coordinating water molecules, one oxygen atom from one bct ligand and two nitrogen atoms from two 1,3-di(1*H*-imidazol-1-yl)propane ligands to

generate a distorted octahedral coordination geometry. The Ni–O bond lengths are 2.041(3)–2.137(3) Å and Ni–N bond length are 2.075(4), and 2.081(4) Å, respectively. The bridging diim ligands also link Ni cations to form a 1D chain [13].

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