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Crystal structure of *catena*-poly[*trans*-tetraaqua (μ_2 -1,1'-(biphenyl-4,4'-diyl)bis(1*H*-imidazol)- $k^2N:N'$)cobalt(II)] dinitrate – 1,1'-(biphenyl-4,4'-diyl)bis(1*H*-imidazol) – water (1/3/2), $C_{72}H_{68}CoN_{18}O_{12}$

Abstract

$C_{72}H_{68}CoN_{18}O_{12}$, triclinic, $P\bar{1}$ (no. 2), $a = 9.2801(10)$ Å, $b = 11.6017(11)$ Å, $c = 16.4968(15)$ Å, $\alpha = 96.294(8)^\circ$, $\beta = 96.371(8)^\circ$, $\gamma = 105.025(9)^\circ$, $V = 1687.0(3)$ Å³, $Z = 1$, $R_{gt}(F) = 0.0631$, $wR_{ref}(F^2) = 0.1043$, $T = 293(2)$ 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.

Table 1: Data collection and handling.

Crystal:	Purple block
Size:	0.25 × 0.23 × 0.20 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.33 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} :	11546, 5939, 0.081
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 3359
$N(param)_{refined}$:	505
Programs:	Bruker [1], SHELX [2]

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Source of material

All chemicals and solvents were of analytical grade and used without further purification. The title compound was prepared by the hydrothermal method. A mixture of $Co(NO_3)_2 \cdot 6H_2O$ (1 mmol, 0.291 g), 1,1'-(biphenyl-4,4'-diyl)bis(1*H*-imidazol) (bibp) (1 mmol, 0.288 g), dimethylformamide (6 ml) and H_2O (2 ml) were placed in a 25 mL Teflon-lined autoclave at 413 K for 72 h, then cooled to room temperature. Purple block crystals were obtained.

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}
Co1	1.000000	1.000000	0.500000	0.0353 (2)
O1	1.0038 (3)	0.8289 (2)	0.44874 (16)	0.0541 (8)
H1A	1.0705 (19)	0.7956 (18)	0.466 (2)	0.081*
H1B	0.9293 (18)	0.7712 (14)	0.4250 (19)	0.081*
O2	1.1532 (3)	1.0772 (2)	0.42165 (13)	0.0459 (7)
H2A	1.158 (4)	1.0397 (18)	0.3752 (8)	0.069*
H2B	1.163 (4)	1.1499 (7)	0.4138 (14)	0.069*
N1	0.8093 (3)	0.9885 (2)	0.41180 (16)	0.0358 (7)
N2	0.6616 (3)	0.9836 (2)	0.29555 (16)	0.0350 (7)
N3	0.7781 (4)	0.2720 (3)	0.4883 (2)	0.0570 (10)
N4	0.6408 (4)	0.2913 (3)	0.3748 (2)	0.0481 (9)
N5	0.3760 (4)	0.3408 (3)	−0.2191 (2)	0.0506 (9)
N6	0.2387 (4)	0.3217 (3)	−0.3417 (2)	0.0698 (12)
N7	0.1667 (4)	0.3067 (3)	0.3836 (2)	0.0612 (10)
N8	0.1175 (3)	0.4093 (3)	0.28392 (19)	0.0450 (8)
C1	0.8038 (4)	0.9973 (3)	0.3322 (2)	0.0389 (10)
H1	0.887689	1.011180	0.304849	0.047*
C2	0.6624 (4)	0.9681 (3)	0.4258 (2)	0.0450 (10)
H2	0.630182	0.957500	0.476524	0.054*
C3	0.5711 (4)	0.9656 (3)	0.3555 (2)	0.0497 (11)
H3	0.467304	0.954003	0.349193	0.060*
C4	0.6161 (4)	0.9879 (3)	0.2102 (2)	0.0334 (9)
C5	0.4714 (4)	0.9269 (3)	0.1737 (2)	0.0441 (10)
H5	0.404050	0.882063	0.203798	0.053*
C6	0.4277 (4)	0.9330 (3)	0.0921 (2)	0.0478 (11)
H6	0.329225	0.893042	0.068277	0.057*
C7	0.5244 (4)	0.9962 (3)	0.04438 (18)	0.0348 (9)
C8	0.6685 (4)	1.0564 (3)	0.0828 (2)	0.0564 (12)
H8	0.736570	1.100478	0.052723	0.068*
C9	0.7140 (4)	1.0527 (3)	0.1652 (2)	0.0542 (12)
H9	0.811462	1.094468	0.189772	0.065*
C10	0.6345 (5)	0.2678 (4)	0.5039 (3)	0.0612 (12)
H10	0.600940	0.258794	0.554485	0.073*
C11	0.5491 (5)	0.2788 (4)	0.4344 (3)	0.0590 (12)
H11	0.447978	0.277956	0.428453	0.071*
C12	0.7769 (4)	0.2861 (3)	0.4109 (3)	0.0542 (11)
H12	0.860263	0.292013	0.383447	0.065*
C13	0.6016 (4)	0.3016 (3)	0.2902 (2)	0.0433 (10)
C14	0.4634 (4)	0.2341 (3)	0.2476 (3)	0.0550 (12)
H14	0.395416	0.183114	0.274212	0.066*
C15	0.4269 (4)	0.2425 (4)	0.1661 (3)	0.0579 (12)
H15	0.332361	0.198385	0.138623	0.069*
C16	0.5262 (4)	0.3146 (3)	0.1234 (2)	0.0430 (10)
C17	0.6656 (4)	0.3813 (3)	0.1673 (2)	0.0539 (11)
H17	0.735348	0.430710	0.140784	0.065*
C18	0.7010 (4)	0.3745 (3)	0.2497 (2)	0.0524 (11)
H18	0.793941	0.420220	0.278205	0.063*
C19	0.4886 (4)	0.3222 (3)	0.0343 (3)	0.0454 (10)
C20	0.3460 (5)	0.2666 (4)	−0.0087 (3)	0.0613 (12)
H20	0.272345	0.224095	0.019095	0.074*
C21	0.3093 (4)	0.2721 (4)	−0.0913 (3)	0.0636 (13)
H21	0.212724	0.232601	−0.118275	0.076*
C22	0.4141 (4)	0.3354 (3)	−0.1338 (2)	0.0461 (10)
C23	0.5564 (4)	0.3932 (4)	−0.0931 (3)	0.0607 (12)
H23	0.628751	0.438122	−0.120528	0.073*

Table 2: (continued)

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
C24	0.5901 (4)	0.3837 (3)	−0.0109 (3)	0.0581 (12)
H24	0.687728	0.421311	0.015320	0.070*
C25	0.2369 (5)	0.3047 (4)	−0.2648 (3)	0.0675 (14)
H25	0.149281	0.271190	−0.243565	0.081*
C26	0.3871 (6)	0.3714 (4)	−0.3473 (3)	0.0792 (15)
H26	0.424473	0.393081	−0.395106	0.095*
C27	0.4713 (5)	0.3843 (4)	−0.2733 (3)	0.0766 (15)
H27	0.575213	0.416802	−0.261092	0.092*
C28	0.1692 (5)	0.4196 (4)	0.4179 (3)	0.0702 (14)
H28	0.188047	0.447873	0.474074	0.084*
C29	0.1398 (5)	0.4853 (4)	0.3572 (3)	0.0636 (13)
H29	0.135770	0.564945	0.363992	0.076*
C30	0.1349 (4)	0.3041 (4)	0.3042 (3)	0.0558 (12)
H30	0.125348	0.236641	0.265719	0.067*
C31	0.0848 (4)	0.4368 (3)	0.2028 (2)	0.0438 (10)
C32	0.0821 (4)	0.5500 (3)	0.1893 (2)	0.0515 (11)
H32	0.103378	0.611364	0.233803	0.062*
C33	0.0486 (4)	0.5747 (3)	0.1112 (2)	0.0512 (11)
H33	0.046078	0.652578	0.103957	0.061*
C34	0.0180 (4)	0.4864 (3)	0.0423 (2)	0.0418 (10)
C35	0.0209 (4)	0.3720 (3)	0.0576 (2)	0.0537 (11)
H35	−0.000031	0.310292	0.013362	0.064*
C36	0.0534 (4)	0.3462 (3)	0.1357 (3)	0.0562 (12)
H36	0.054475	0.268310	0.143628	0.067*
O3	0.0674 (4)	0.8692 (4)	0.1503 (3)	0.1113 (15)
O4	0.1134 (5)	1.0287 (4)	0.2370 (3)	0.1302 (19)
O5A ^a	0.0664 (13)	1.0425 (10)	0.1122 (7)	0.113 (4)
N9 ^a	0.0805 (10)	0.9815 (12)	0.1664 (9)	0.083 (3)
O1WA ^a	0.1943 (11)	0.8175 (8)	0.3102 (6)	0.107 (3)
H1WA ^a	0.146102	0.839748	0.347110	0.161*
H1WB ^a	0.150363	0.821427	0.262940	0.161*
O5B ^a	0.1640 (15)	0.8836 (13)	0.2780 (8)	0.161 (6)
N9B ^a	0.1127 (11)	0.9224 (11)	0.2226 (8)	0.086 (3)
O1WB ^a	0.0484 (14)	1.0747 (9)	0.0584 (6)	0.118 (4)
H1WC ^a	0.003260	1.046526	0.009772	0.177*
H1WD ^a	0.111260	1.035676	0.071562	0.177*

^a Occupancy: 0.5

Experimental details

H atoms bound to C atoms were placed in calculated positions and refined as riding on their parent atoms, with C–H = 0.93 Å and with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$. The O-bound H atoms were located in a difference Fourier map and refined with a distance restraint of O–H = 0.85 Å, and with $U_{\text{iso}}(\text{H})$ set to 1.5 $U_{\text{eq}}(\text{O})$. The uncoordinated water molecules and the nitrate anions are disordered over two positions in this structure.

Comment

Imidazole is a versatile *N*-donor organic ligand in construction of coordination polymers because of the potential

coordination sites [3–5]. As a imidazole derivative, 1,1'-(biphenyl-4,4'-diyl)bis(1*H*-imidazol) (bibp) was widely used to synthesise coordination polymers due to different coordination modes of the bibp ligand [6–11].

The asymmetric unit of the crystal structure consists of half of a Co(II) center located on a crystallographic inversion center, one half of a bibp ligand, two coordinated water molecules, one and a half free bibp ligands (see the Figure). The nitrate counter anion and one more uncoordinated water molecule are both disordered and not shown in the Figure. The Co(II) ion is six-coordinated by four O atoms from two pairs of symmetry related water molecules (Co–O = 2.081(2)–2.120(2) Å), and two N atoms from two symmetry related bibp ligands (Co–N = 2.129(3) Å), forming a distorted octahedral geometry. The coordinated bibp ligands link adjacent Co(II) ions into a polymeric chain structure with the Co–Co distance of 18.008 Å along the *c* axis. In the crystal structure, O–H···O and O–H···N hydrogen bonds link molecules and ions into a three-dimensional supermolecular framework.

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