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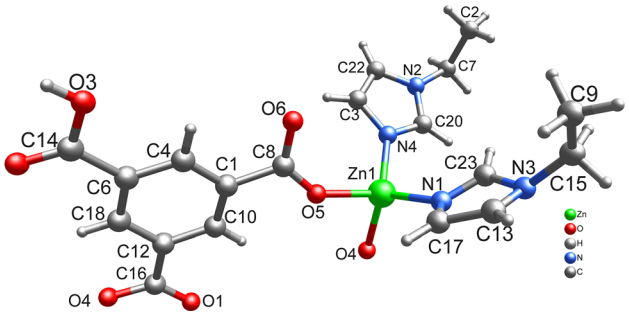
The crystal structure of *catena*-poly [bis(1-ethylimidazole-k^1*N*)-(μ_2-benzene-1-carboxyl-3,5-dicarboxylato-κ^2*O*, *O*′)zinc(II)], $C_{19}H_{20}N_4O_6Zn$



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Abstract

$C_{19}H_{20}N_4O_6Zn$, monoclinic, $P2_1/n$ (no. 14), a = 11.7411(7) Å, b = 13.7831(9) Å, c = 13.5425(10) Å, β = 97.564 (2)°, V = 2172.5(3) Å^3, Z = 4, $R_{gt}(F)$ = 0.0645, $wR_{ref}(F^2)$ = 0.1643, T = 298 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:	Colourless block
Size:	0.24 × 0.22 × 0.20 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	1.17 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	25.1°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	18959, 3838, 0.083
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 2571
$N(param)_{refined}$:	311
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

1 Source of materials

The title compound was obtained by a reaction of zinc sulfate heptahydrate (0.287 g, 1 mmol), 1,3,5-benzenetricarboxylic acid (0.42 g, 2 mmol), 1-ethylimidazole (0.6 mL), in mixture solution of 5 mL N, N′-dimethylformamide and 2.5 mL dimethyl sulfoxide, which was heated in a 25 mL steel autoclave and then was heated to 423 K for 6 h. After the mixture solutions were cooled to room temperature, colorless block crystals were collected from the mother liquor.

2 Experimental details

The structure was solved by Direct Methods with the SHELXS-2018 program. All H-atoms from C and N atoms were positioned with idealized geometry and refined isotropically (U_{iso}(H) = 1.2U_{eq}(C)) using a riding model. The four C atoms of 1-ethylimidazole are processed with part instruction to model the disorder.

3 Comment

Considerable effort has been devoted to the assembly of zinc-1,3,5-benzenetricarboxylic acid-based coordination polymers and many crystal structures are reported [5–8]. Although zinc-1,3,5-benzenetricarboxylic acid and different imidazole-containing ligands have been used as co-ligands to construct zinc coordination polymers [9–15], to the best of our knowledge, there has not been reported any result of zinc-1,3,5-benzenetricarboxylic acid and neutral N-containing ligand 1-ethylimidazole.

Single X-ray diffraction study shows that the title compound crystallizes in the monoclinic space group $P2_1/n$ (no. 14). The asymmetrical unit in title compound contains one Zn(II) cation, one benzene-1-carboxyl-3,5-dicarboxylate dianion (bcd) and two coordinated 1-ethylimidazole ligands.

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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}
Zn1	0.48561 (5)	0.76440 (4)	0.72839 (6)	0.0398 (3)
O1	0.1588 (3)	0.4217 (3)	0.7613 (3)	0.0527 (12)
O2	0.5654 (3)	0.1140 (3)	0.7721 (4)	0.0703 (15)
O3	0.7102 (3)	0.2117 (3)	0.7568 (4)	0.0672 (15)
H3	0.744914	0.160348	0.753970	0.101*
O4	0.1813 (3)	0.2632 (3)	0.7803 (3)	0.0481 (11)
O5	0.4964 (3)	0.6236 (3)	0.7240 (4)	0.0700 (15)
O6	0.6692 (3)	0.5602 (3)	0.7541 (4)	0.0621 (14)
N1	0.5453 (4)	0.8227 (4)	0.6124 (4)	0.0552 (12)
N2	0.6529 (6)	0.9137 (5)	0.9665 (5)	0.0820 (16)
N3	0.6419 (6)	0.9141 (5)	0.5213 (5)	0.0802 (15)
N4	0.5711 (4)	0.8132 (4)	0.8536 (4)	0.0528 (12)
C1	0.5111 (4)	0.4561 (3)	0.7488 (4)	0.0286 (10)
C2	0.8053 (9)	1.0279 (9)	1.0019 (8)	0.147 (4)
H2A	0.850073	0.971592	1.023284	0.220*
H2B	0.834403	1.082993	1.040569	0.220*
H2C	0.810204	1.039872	0.932728	0.220*
C3	0.6292 (7)	0.7631 (6)	0.9315 (6)	0.0791 (18)
H3A	0.632487	0.695816	0.936403	0.095*
C4	0.5787 (4)	0.3738 (3)	0.7507 (4)	0.0308 (11)
H4	0.656485	0.379486	0.745073	0.037*
C5[a]	0.7086 (8)	1.0023 (7)	0.4988 (8)	0.0886 (19)
H5A[a]	0.672104	1.033846	0.438834	0.106*
H5B[a]	0.712891	1.048108	0.553492	0.106*
C6	0.5317 (4)	0.2832 (3)	0.7609 (4)	0.0305 (10)
C7	0.6839 (8)	1.0111 (7)	1.0158 (7)	0.101 (2)
H7A	0.635134	1.062153	0.984323	0.121*
H7B	0.675526	1.008960	1.086059	0.121*
C8	0.5638 (4)	0.5539 (3)	0.7414 (4)	0.0352 (12)
C9[b]	0.848 (2)	0.8928 (18)	0.4688 (18)	0.081 (4)
H9A[b]	0.895165	0.884089	0.531771	0.121*
H9B[b]	0.890698	0.928014	0.424551	0.121*
H9C[b]	0.826798	0.830517	0.440451	0.121*
C10	0.3948 (4)	0.4470 (3)	0.7543 (4)	0.0298 (11)
H10	0.348828	0.502201	0.751113	0.036*
C11[c]	0.5090 (13)	0.8150 (11)	0.5091 (11)	0.080 (2)
H11[c]	0.451016	0.773716	0.480848	0.096*
C12	0.3459 (4)	0.3564 (3)	0.7644 (4)	0.0289 (10)
C13[d]	0.6226 (18)	0.8296 (16)	0.4773 (17)	0.078 (2)
H13[d]	0.648673	0.810779	0.418256	0.093*
C14	0.6027 (4)	0.1938 (4)	0.7644 (4)	0.0364 (12)
C15[e]	0.752 (2)	0.944 (3)	0.482 (3)	0.076 (2)
H15A[e]	0.726067	0.971546	0.417557	0.091*
H15B[e]	0.780108	0.997631	0.524457	0.091*
C16	0.2200 (4)	0.3484 (4)	0.7692 (4)	0.0344 (11)
C17[d]	0.5564 (19)	0.7726 (17)	0.5337 (16)	0.078 (2)
H17[d]	0.526591	0.711176	0.517726	0.093*
C18	0.4156 (4)	0.2751 (3)	0.7684 (4)	0.0314 (11)
H18	0.384013	0.214188	0.776215	0.038*
C19[f]	0.8243 (9)	0.9682 (10)	0.4849 (10)	0.090 (3)
H19A[f]	0.863716	0.946090	0.547457	0.135*
H19B[f]	0.866675	1.020516	0.460297	0.135*
H19C[f]	0.818060	0.915759	0.437806	0.135*
C20	0.5870 (7)	0.9045 (5)	0.8788 (6)	0.0723 (16)
H20	0.556285	0.956452	0.840302	0.087*
C21[c]	0.5688 (12)	0.8745 (10)	0.4568 (11)	0.076 (2)
H21[c]	0.558672	0.884483	0.388245	0.092*
C22	0.6800 (7)	0.8236 (6)	0.9987 (6)	0.0814 (18)
H22	0.726074	0.806948	1.057510	0.098*
C23	0.6114 (7)	0.8972 (6)	0.6127 (6)	0.0771 (15)
H23	0.635292	0.934674	0.668686	0.093*

[a]Occupancy: 0.8, [b]Occupancy: 0.35, [c]Occupancy: 0.6, [d]Occupancy: 0.4, [e]Occupancy: 0.2, [f]Occupancy: 0.65.

The Zn cation is tetrahedrally coordinated by two O atoms of *bcd* and two N atoms from two 1-ethylimidazole ligands, which is common in such zinc complexes [9–15].

The Zn-O, Zn-N bond distances are 1.9426–1.9478 Å and 1.9716–1.9733 Å, respectively. The bond angles around the central Zn vary from 93.4° to 116.7°, indicating that the tetrahedron is slightly distorted. All the bond lengths and angles are comparable with that observed in related zinc(II) complexes [9–15]. The results show that two protons of 1,3,5-benzenetricarboxylic acid are removed for the balance charge and bridged two Zn cation, which leads to the formation of 1D chains. It is worth noting that the 1D chains are different from the previous reports of zinc-1,3,5-benzenetricarboxylic acid-based chains [8–10]. A 2D layered structure is constructed by intermolecular hydrogen bond of oxygen atoms of the carboxyl groups between chains.

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