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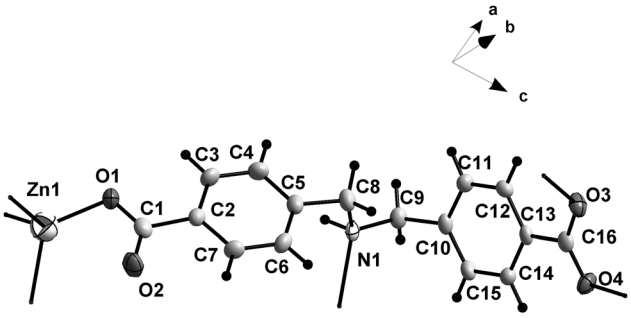
The crystal structure of poly[(μ_4-4,4′-(azanediylbis(methylene))dibenzoato-$\kappa^4O:N:O':O''$) zinc(II)], $C_{16}H_{13}NO_4Zn$



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Abstract

$C_{16}H_{13}NO_4Zn$, triclinic, $P\bar{1}$ (no. 2), a = 7.5223(6) Å, b = 8.0977(8) Å, c = 11.7739(11) Å, α = 76.410(8)°, β = 75.495(8)°, γ = 75.902(8)°, V = 661.76(11) Å^3, Z = 2, $R_{gt}(F)$ = 0.0532, w$R_{ref}(F^2)$ = 0.1041, T = 293(2) K.

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A part of the title coordination-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.

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.30 × 0.20 × 0.20 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.87 mm^{-1}
Diffractometer, scan mode:	φ and ω
θ_{max}, completeness:	26.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	5365, 2700, 0.043
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2146
$N(param)_{refined}$:	199
Programs:	CrysAlis^{PRO} [1], Diamond [2], SHELX [3, 4]

Source of material

All reagents and chemicals were purchased from commercial sources and used without further purification. The starting material 4,4′-(azanediylbis(methylene))dibenzoic acid was synthesized following the literature procedures [5]. A mixture of $Zn(NO_3)_2{\cdot}6H_2O$ (0.05 mmol), 4,4′-(azanediylbis(methylene)) dibenzoic acid (0.05 mmol), *N*,*N*-dimethylacetamide (0.5 mL), ethanol(3 mL), and H_2O (3 mL) was sealed in a 20 mL vial and heated at 363 K for two days. Colorless block crystals were collected. **IR spectra** (potassium bromide pellet) were recorded on a Nicolet 6700. IR (ν/cm^{-1}): 3433, 3238, 3049, 2974, 2929, 2869, 1612, 1597, 1545, 1509, 1403, 1371, 1356, 1308, 1200, 1186, 1066, 1024, 908, 881, 860, 816, 801, 777, 768, 719, 707, 669, 575, 540, 464.

Experimental details

H atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2 U_{eq}(C). The nitrogen-bound H atoms were located on a difference Fourier map.

Comment

In recent years, 4,4′-(azanediylbis(methylene))dibenzoic acid and its analogues have been widely applied in the formation of zinc coordination polymers [6–9]. Some zinc complexes have drawn tremendous attention because of their potential applications in gas separation [8] and chemical sensor [9]. To further explore this area, we used 4,4′-(azanediylbis(methylene))dibenzoic acid to prepare a new zinc coordination polymer.

The asymmetric unit consists of one zinc cation and one 4,4′-(azanediylbis(methylene))dibenzoate ligand.

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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	0.39487 (7)	−0.04929 (6)	0.17140 (4)	0.02808 (17)
O1	0.5265 (4)	0.0752 (4)	0.2345 (2)	0.0329 (7)
O2	0.2504 (4)	0.1432 (4)	0.3528 (3)	0.0415 (8)
O3	0.7020 (4)	0.8984 (3)	0.9672 (2)	0.0315 (7)
O4	0.5634 (4)	0.7367 (3)	1.1270 (2)	0.0312 (7)
N1	0.8378 (4)	0.1459 (4)	0.7303 (3)	0.0242 (8)
H1	0.904927	0.059783	0.680304	0.029*
C1	0.4206 (6)	0.1377 (5)	0.3234 (4)	0.0278 (10)
C2	0.5157 (6)	0.2007 (5)	0.3991 (3)	0.0245 (9)
C3	0.7092 (6)	0.1910 (5)	0.3717 (4)	0.0321 (10)
H3	0.779258	0.157377	0.300797	0.039*
C4	0.7978 (6)	0.2305 (5)	0.4483 (4)	0.0334 (10)
H4	0.926755	0.223642	0.428774	0.040*
C5	0.6950 (6)	0.2805 (5)	0.5543 (3)	0.0277 (10)
C6	0.5020 (6)	0.2985 (5)	0.5796 (3)	0.0287 (10)
H6	0.431327	0.338737	0.648197	0.034*
C7	0.4142 (6)	0.2567 (5)	0.5031 (3)	0.0294 (10)
H7	0.284842	0.266442	0.521904	0.035*
C8	0.7944 (6)	0.3092 (5)	0.6429 (4)	0.0325 (10)
H8A	0.909678	0.347862	0.600660	0.039*
H8B	0.715657	0.398991	0.685310	0.039*
C9	0.9766 (6)	0.1532 (5)	0.8002 (3)	0.0270 (9)
H9A	1.095361	0.165128	0.746140	0.032*
H9B	0.996492	0.045570	0.856850	0.032*
C10	0.9098 (5)	0.3033 (5)	0.8667 (3)	0.0238 (9)
C11	0.9651 (6)	0.4602 (5)	0.8147 (3)	0.0261 (9)
H11	1.052979	0.468940	0.743330	0.031*
C12	0.8907 (5)	0.6027 (5)	0.8680 (3)	0.0246 (9)
H12	0.927627	0.707377	0.831274	0.030*
C13	0.7622 (5)	0.5936 (5)	0.9750 (3)	0.0209 (8)
C14	0.7127 (6)	0.4337 (5)	1.0307 (3)	0.0251 (9)
H14	0.629213	0.423568	1.103915	0.030*
C15	0.7883 (6)	0.2906 (5)	0.9767 (3)	0.0262 (9)
H15	0.756932	0.184162	1.014963	0.031*
C16	0.6699 (5)	0.7527 (5)	1.0257 (4)	0.0234 (9)

U_{eq} is equivalent isotropic displacement parameters. U_{iso}* is isotropic displacement parameters. So '*' is U_{iso}*.

The zinc(II) ion is surrounded by one nitrogen atom and three oxygen atoms from four different ligands. Its coordination geometry is a slightly distorted tetrahedron. The Zn—O bond lengths range from 1.983(3) to 1.977(3) Å. The Zn—N bond is 2.059(3) Å, and are in the expected ranges [10]. Adjacent ligands have intramolecular N1—H1···O2 hydrogen bond of length 2.384 Å. The zinc coordination polymer is extended to a two dimensional layer along the *bc* plane. There are weak $\pi \cdots \pi$ interactions between C10—C15 phenyl rings from adjacent layers. The centroid-centroid distance is 3.745(2) Å. Together with the $\pi \cdots \pi$ stacking interactions a three-dimensional network is obtained.

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