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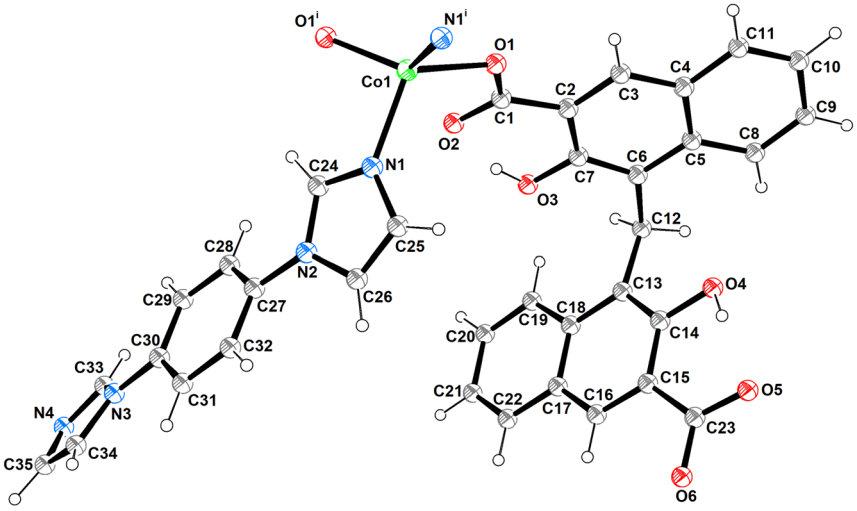
The crystal structure of poly[bis(μ_2-1,4-bi(1-imidazolyl)benzene-κ^2N:N')bis(μ_2-4,4′-methylenebis(3-hydroxy-2-naphthoate)-κ^2O:O')cobalt(II)], $C_{35}H_{24}CoN_4O_6$



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Abstract

$C_{35}H_{24}CoN_4O_6$, monoclinic, $P2_1/n$ (no. 14), a = 11.444(2) Å, b = 11.648(2) Å, c = 21.478(4) Å, β = 97.05(3)°, V = 2841.5(10) Å^3, Z = 4, $R_{gt}(F)$ = 0.0849, $wR_{ref}(F^2)$ = 0.1393, T = 153(2) K.

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A part of the title crystal 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:	Purple block
Size:	0.10 × 0.08 × 0.06 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.66 mm^{-1}
Scan mode:	φ and ω
θ_{max}, completeness:	25.0°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	15,908, 4985, 0.071
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 4250
$N(param)_{refined}$:	415
Programs:	CrystalClear [1], Olex2 [2], SHELX [3, 4], WinGX/ORTEP [5]

1 Source of material

All the reagents and solvents were used as obtained without further purification. $CoCl_2{\cdot}6H_2O$ (0.1 mmol, 23.8 mg), pamoic acid (H_2PA) (0.05 mmol, 19.4 mg) and 1,4-bi(1-imidazolyl) benzene (BIB) (0.05 mmol, 10.5 mg) were completely dissolved in 12 mL DMF/H_2O (1/2, v/v). The mixture was transferred to a 25 mL Teflon-lined stainless steel autoclave and was heated under autogenous pressure at 393 K for 3 days.

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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}
Co1	0.94307 (6)	0.56909 (6)	0.28832 (3)	0.0270 (2)
O1	0.8557 (3)	0.7148 (3)	0.29273 (17)	0.0357 (9)
O2	0.8421 (3)	0.6766 (3)	0.39323 (16)	0.0317 (8)
O3	0.6759 (3)	0.7662 (3)	0.44504 (15)	0.0301 (8)
H3	0.735006	0.728312	0.437537	0.036*
O4	0.3016 (3)	0.8293 (3)	0.34607 (16)	0.0323 (8)
H4	0.253893	0.785494	0.324848	0.039*
O5	0.1261 (3)	0.7060 (3)	0.30042 (16)	0.0360 (9)
O6	0.0908 (3)	0.5399 (3)	0.34381 (17)	0.0357 (9)
N1	0.8587 (3)	0.4310 (3)	0.32023 (18)	0.0276 (9)
N2	0.8301 (3)	0.2894 (3)	0.38500 (18)	0.0243 (9)
N3	0.8946 (3)	−0.0081 (3)	0.59390 (18)	0.0266 (9)
N4	0.9256 (3)	−0.0635 (4)	0.69297 (18)	0.0292 (10)
C1	0.8126 (4)	0.7332 (4)	0.3437 (2)	0.0292 (12)
C2	0.7215 (4)	0.8255 (4)	0.3436 (2)	0.0246 (11)
C3	0.7011 (4)	0.9008 (4)	0.2942 (2)	0.0276 (12)
H3A	0.749782	0.898284	0.261496	0.033*
C4	0.6082 (4)	0.9824 (4)	0.2914 (2)	0.0244 (11)
C5	0.5290 (4)	0.9790 (4)	0.3379 (2)	0.0227 (11)
C6	0.5504 (4)	0.9001 (4)	0.3891 (2)	0.0223 (11)
C7	0.6485 (4)	0.8310 (4)	0.3921 (2)	0.0241 (11)
C8	0.4341 (4)	1.0587 (4)	0.3309 (2)	0.0284 (11)
H8	0.377922	1.057282	0.359924	0.034*
C9	0.4220 (4)	1.1373 (4)	0.2833 (2)	0.0312 (12)
H9	0.357363	1.188980	0.279888	0.037*
C10	0.5034 (5)	1.1431 (4)	0.2393 (2)	0.0341 (13)
H10	0.495722	1.200221	0.207521	0.041*
C11	0.5932 (4)	1.0660 (4)	0.2430 (2)	0.0320 (12)
H11	0.646855	1.068030	0.212549	0.038*
C12	0.4720 (4)	0.8906 (4)	0.4410 (2)	0.0265 (11)
H12A	0.520853	0.902030	0.481890	0.032*
H12B	0.413070	0.953228	0.435910	0.032*
C13	0.4078 (4)	0.7771 (4)	0.4426 (2)	0.0229 (11)
C14	0.3211 (4)	0.7514 (4)	0.3941 (2)	0.0244 (11)
C15	0.2514 (4)	0.6506 (4)	0.3933 (2)	0.0253 (11)
C16	0.2708 (4)	0.5762 (4)	0.4422 (2)	0.0313 (12)
H16	0.224983	0.508108	0.441712	0.038*
C17	0.3566 (4)	0.5971 (4)	0.4936 (2)	0.0292 (12)
C18	0.426 (4)	0.6984 (4)	0.4945 (2)	0.0284 (12)
C19	0.5119 (5)	0.7164 (5)	0.5472 (2)	0.0321 (12)
H19	0.559574	0.783365	0.549212	0.039*
C20	0.5267 (5)	0.6386 (5)	0.5951 (3)	0.0410 (14)
H20	0.584983	0.651984	0.629776	0.049*
C21	0.4568 (6)	0.5387 (5)	0.5937 (3)	0.0487 (16)
H21	0.467320	0.485537	0.627433	0.058*
C22	0.3749 (5)	0.5192 (5)	0.5441 (3)	0.0403 (14)
H22	0.328506	0.451510	0.543246	0.048*
C23	0.1520 (4)	0.6319 (4)	0.3422 (2)	0.0293 (12)
C24	0.9033 (4)	0.3734 (4)	0.3704 (2)	0.0302 (12)
H24	0.977802	0.389066	0.393529	0.036*
C25	0.7496 (4)	0.3824 (4)	0.3019 (2)	0.0307 (12)
H25	0.695611	0.407128	0.267336	0.037*
C26	0.7314 (4)	0.2947 (5)	0.3404 (2)	0.0326 (13)
H26	0.664301	0.246019	0.337613	0.039*
C27	0.8500 (4)	0.2126 (4)	0.4373 (2)	0.0270 (11)
C28	0.9204 (4)	0.2441 (4)	0.4915 (2)	0.0308 (12)
H28	0.957696	0.317114	0.493846	0.037*
C29	0.9371 (4)	0.1712 (4)	0.5420 (2)	0.0312 (12)
H29	0.987050	0.193554	0.578634	0.037*
C30	0.8818 (4)	0.0652 (4)	0.5400 (2)	0.0242 (11)
C31	0.8105 (4)	0.0330 (4)	0.4863 (2)	0.0311 (12)
H31	0.771326	−0.039043	0.484564	0.037*
C32	0.7960 (4)	0.1063 (4)	0.4346 (2)	0.0321 (12)
H32	0.748547	0.083026	0.397342	0.039*
C33	0.9292 (4)	0.0237 (4)	0.6544 (2)	0.0284 (12)
H33	0.952836	0.099221	0.667033	0.034*
C34	0.8663 (5)	−0.1236 (4)	0.5946 (2)	0.0341 (13)
H34	0.838415	−0.170148	0.559556	0.041*
C35	0.8867 (5)	−0.1567 (5)	0.6555 (2)	0.0358 (13)
H35	0.875934	−0.232353	0.670349	0.043*

After the reactant was slowly cooled to room temperature, purple block crystals of the title compound were obtained, yielding 39.8 % (based on the H_2PA).

2 Experimental details

A suitable single crystal was mounted onto the end of a thin glass fiber using Fomblin oil. An absorption correction was applied [1]. The structure solution and the refinement were successfully carried out using SHELX program system [2–4]. H atoms were treated as riding, with C–H distances of 0.95 Å (aromatic); U_{iso} (H) = 1.2 U_{eq}. H atoms bonded to O atoms were initially found in different maps, and refined with constraint of O–H = 0.84 Å and U_{iso} (H) = 1.2 U_{eq} (O). The figure was created using the ORTEP-3 software [5].

3 Comment

In the past few decades, coordination polymer materials have been widely used in various fields such as catalysis, gas separation and storage, sensing, and proton conduction due to their unique crystalline porous nature, flexible tailoring characteristics, and extremely high surface area [6–8]. The design and synthesis of novel coordination polymer materials are beneficial for the research of fluorescent sensing materials [9]. In the course of our efforts to explore new cobalt-containing coordination polymers architectures using PA^{2-} and BIB ligands, the title compound was successfully synthesized and determined by single-crystal X-ray analysis.

The asymmetric unit comprises one Co(II) cation, one PA^{2-} anion and one BIB ligand. Each Co(II) center is four-coordinated with a distorted tetrahedral environment by two N atoms and two O atoms. The bond lengths and angles within structure are unexceptional and lie within the expected ranges. In details, the Co–N bond lengths are 2.034(4) and 2.038(4) Å. The Co–O bond lengths are 1.974(3) and 1.978(3) Å. The bond angles around Co(II) range from 93.84(15)° to 124.98(16)°. All bond lengths and angles are in the expected ranges [10–12].

For the PA^{2-} ligand, the hydrogen atom of the phenolic hydroxy group is still retained, only one of the carboxylate oxygen atoms is coordinated with the Co(II) ion. Although there are no strong intermolecular hydrogen bonds in the structure, the uncoordinated hydroxyl oxygen atoms (O3 and O4) of the PA^{2-} ligand are protonated and form strong intramolecular hydrogen bonds with the carboxyl oxygen atoms (O2 and O5) within the same molecule. The distances of O···H and the bond angles of O–H···O are 1.7484(36) Å and 1.7559(34) Å, 157.01(26)° and 160.881(262)°, respectively. The PA^{2-} ligand bridge adjacent Co(II) cations to form chains through O atoms in one direction, whereas the BIB ligand bridge adjacent Co(II) cations to form chains through N atoms in the other direction. The chains are further extended and finally resulted in a 2D network.

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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References

1. Rigaku. *CrystalClear*; Rigaku Corporation: The Woodlands, Texas, USA, 2008.
2. Dolomanov O. V., Bourhis L. J., Gildea R. J., Howard J. A. K., Puschmann H. OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* 2009, *42*, 339–341.
3. Sheldrick G. M. Crystal structure refinement with *SHELXL*. *Acta Crystallogr.* 2015, *C71*, 3–8.
4. Sheldrick G. M. *SHELXTL* – integrated space-group and crystal-structure determination. *Acta Crystallogr.* 2015, *A71*, 3–8.
5. Louis J. F. *WinGX* and *ORTEP* for *Windows*: an update. *J. Appl. Crystallogr.* 2012, *45*, 849–854.
6. Rath B. B., Vittal J. J. Dynamic effects in crystalline coordination polymers. *CrystEngComm* 2021, *23*, 5738–5752.
7. Fernandez-Bartolome E., Gamonal A., Santos J., Khodabakhshi S., Rodríguez-Sánchez E., Carolina Sañudo E., Martín N., Costa J. S. Playing with the weakest supramolecular interactions in a 3D crystalline hexakis [60] fullerene induces control over hydrogenation selectivity. *Chem. Sci.* 2021, *12*, 8682–8688.
8. Yang Y., Wang K. Z., Yan D. Smart luminescent coordination polymers towards multi–mode logic gates: time-resolved, tribochromic and excitation-dependent fluorescence/phosphorescence emission. *ACS Appl. Mater. Interfaces* 2017, *9*, 17399–17407.
9. Chu J.-F., Wang S.-Y., Liu J.-C., Hu H.-B., Xu Q.-X. Two cadmium(II) coordination polymers based on pamoic acid and different polydentate N-donor ligands: syntheses, crystal structures, and fluorescence properties. *Z. Anorg. Allg. Chem.* 2020, *646*, 420–425.
10. Shi X.-M., Li M.-X., He X., Liu H.-J., Shao M. Crystal structures and properties of four coordination polymers constructed from flexible pamoic acid. *Polyhedron* 2010, *29*, 2075–2080.
11. Zhang L.-N., Sun X.-L., Du C.-X., Hou H.-W. Structural diversity and fluorescent properties of new metal-organic frameworks constructed from pamoic acid and different N-donor ligands. *Polyhedron* 2014, *72*, 90–95.
12. Bu X., Wei Z., Ren S. Synthesis, structures and magnetic properties of two isomeric coordination polymers constructed from pamoic acid and 1,2-di(4-pyridyl)ethane. *J. Coord. Chem.* 2015, *68*, 471–478.