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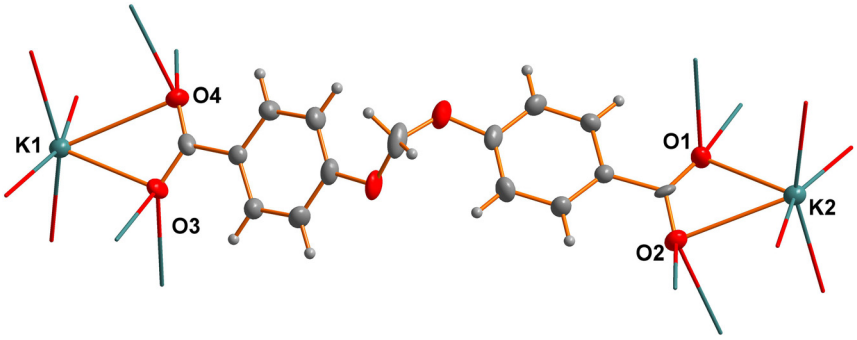
Crystal structure of poly[μ_{10}-4,4′-methylene-bis(oxy)benzoatodipotassium], $C_{15}H_{10}K_2O_6$



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Abstract

$C_{15}H_{10}K_2O_6$, orthorhombic, $Pna2_1$ (no. 33), a = 11.4410(13) Å, b = 3.9881(5) Å, c = 32.465(4) Å, V = 1481.3(3) Å^3, Z = 4, $R_{gt}(F)$ = 0.0356, w$R_{ref}(F^2)$ = 0.0918, T = 296.15 K.

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The molecular 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.

1 Source of materials

To a solution of potassium hydroxide (20 ml, 2 M), 4,4′-methylenebis(oxy)benzoic acid was added to form a transparent solution. The corresponding single crystals were obtained within a week by a slow evaporation.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.10 × 0.10 × 0.10 mm
Wavelength:	MoKα radiation (0.71073 Å)
μ:	0.67 mm^{-1}
Diffractometer, scan mode:	φ and ω
θ_{max}, completeness:	31.0°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	8995, 3943, 0.028
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 $\sigma(I_{obs})$, 3446
$N(param)_{refined}$:	208
Programs:	Bruker [1], Olex2 [2], Shelx [3]

2 Experimental details

The structure was solved by Direct Methods using the Shelx-97 program incorporated into Olex2. All the hydrogen atoms were included in geometric positions and given thermal parameters equivalent to 1.2 times those of the atom to which they were attached [1–3].

3 Comment

The conformationally flexible carboxylates with long linear backbones are popular bridging ligands for the assembly of the entangled architectures, such as interpenetrations, catenanes and rotaxanes. The 4,4′-methylenebis(oxy)benzoic acid is characteristic of a segment of –O–C–O– chain, which endows the two terminal carboxylate groups with more flexibility for those entangled architectures [4–8]. In the asymmetric unit K1 and K2 ions show a similar irregular KO_6 coordination geometry with two O atoms from one carboxylate group and other four O atoms from four distinct

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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}
K1	0.31723 (9)	0.2140 (2)	0.96064 (3)	0.0273 (2)
K2	0.43387 (10)	0.27688 (19)	0.39319 (3)	0.0289 (3)
O1	0.3429 (4)	0.1983 (8)	0.47006 (13)	0.0319 (8)
O2	0.5327 (4)	0.2959 (8)	0.48088 (14)	0.0328 (8)
O3	0.4056 (4)	0.2037 (8)	0.88413 (12)	0.0316 (8)
O4	0.2172 (3)	0.2790 (6)	0.87121 (15)	0.0303 (8)
O9	0.4504 (4)	0.7168 (8)	0.69968 (14)	0.0382 (10)
O14	0.2979 (4)	0.7139 (8)	0.65189 (15)	0.0418 (11)
C1	0.4241 (4)	0.2814 (8)	0.49250 (19)	0.0184 (9)
C2	0.3970 (3)	0.3912 (9)	0.53596 (13)	0.0238 (7)
C3	0.2828 (3)	0.3572 (11)	0.55058 (14)	0.0296 (7)
H3	0.2259	0.2617	0.5338	0.035*
C4	0.2535 (3)	0.4638 (10)	0.58964 (13)	0.0331 (8)
H4	0.1771	0.4412	0.5989	0.040*
C5	0.3371 (4)	0.6039 (10)	0.61498 (13)	0.0309 (8)
C6	0.4525 (3)	0.6249 (12)	0.60203 (14)	0.0351 (9)
H6	0.5101	0.7057	0.6196	0.042*
C7	0.4802 (3)	0.5229 (10)	0.56232 (13)	0.0302 (8)
H7	0.5569	0.5437	0.5532	0.036*
C8	0.3729 (5)	0.9123 (8)	0.67681 (17)	0.0447 (8)
H8A	0.4170	1.0639	0.6594	0.054*
H8B	0.3260	1.0468	0.6954	0.054*
C9	0.4100 (3)	0.6051 (9)	0.73837 (13)	0.0302 (8)
C10	0.4950 (3)	0.4719 (11)	0.76385 (14)	0.0339 (8)
H10	0.5717	0.4532	0.7546	0.041*
C11	0.4659 (3)	0.3665 (11)	0.80317 (13)	0.0301 (7)
H11	0.5235	0.2782	0.8202	0.036*
C12	0.3517 (3)	0.3904 (8)	0.81760 (13)	0.0234 (7)
C13	0.2676 (3)	0.5173 (10)	0.79102 (13)	0.0302 (8)
H13	0.1905	0.5335	0.7999	0.036*
C14	0.2961 (3)	0.6204 (11)	0.75149 (13)	0.0333 (8)
H14	0.2382	0.6996	0.7339	0.040*
C15	0.3177 (4)	0.2824 (9)	0.8609 (2)	0.0263 (12)

carboxylate groups. K1–O4 and K2–O2 bond lengths are 3.132(5) and 3.064(5) Å, respectively. Other K–O distances (K1–O1^1, K1–O1^2, K1–O2^3, K1–O2^4, K1–O3, K2–O3^5, K2–O3^6, K2–O4^7, K2–O4^8, K2–O1) (symmetric codes: ^{1}1/2–X, 1/2 + Y, 1/2 + Z; ^{2}1/2–X, –1/2 + Y, 1/2 + Z; ^{3}1–X,–Y, 1/2 + Z; ^{4}1–X, 1–Y, 1/2 + Z; ^{5}1–X, 1–Y, –1/2 + Z; ^{6}1–X, –Y, –1/2 + Z; ^{7}1/2–X, 1/2 + Y, –1/2 + Z; ^{8}1/2–X, –1/2 + Y, –1/2 + Z) range between 2.680(4) and 3.132(5) Å [9]. Apart from the chelation with one K ion, each carboxylate group bridges additional four K ions. Thus, each 4,4′-methylenebis(oxy)benzoic acid serves as m_{10} linkers, which results in a three-dimensional architecture. Notably, there are extensive weak inter-ring π···π interactions among the 4,4′-methylenebis(oxy)benzoate molecules (centroid-centroid distance: 3.998 Å).

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

References

1. Bruker. *SAINT and SADABS*; Bruker AXS Inc.: Madison, Wisconsin, USA, 2000.
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. Manna P., Tripuramallu B. K., Bommakanti S., Das S. K. Synthesis, characterization and magnetism of metal–organic compounds: role of the positions of the coordinating groups of a meso-flexible ligand in placing anisotropy to exhibit spin-canting behaviour. *Dalton Trans*. 2015, *44*, 2852–2864.
5. Gao Y. P., Guo L., Lv Y. H., Dong W., Jia M., Chang F. A series of 1-D, 2-D and 3-D coordination polymers self-assembled from a flexible dicarboxylate and mixed N-donor ligands: syntheses, structural diversity, and luminescent properties. *J. Coord. Chem.* 2016, *69*, 3745–3761.
6. Zhou M. L., Zhang S. T., Wu Y. B., Liu Y. Two three-fold interpenetrating Co/Cd architectures based on right-and left-helical chains. *Chin. J. Struct. Chem.* 2020, *39*, 1483–1488.
7. Lan Y. Q., Li S. L., Qin J. S., Du D. Y., Wang X. L., Su Z. M., Fu Q. Self-assembly of 2D→2D interpenetrating coordination polymers showing polyrotaxane-and polycatenane-like motifs: influence of various ligands on topological structural diversity. *Inorg. Chem.* 2008, *47*, 10600–10610.
8. Guo L., Gao Y. P., Lv Y. H., Wang L., Han Y., Chang F. A series of 2D and 3D coordination polymers containing multicore Mn-carboxyl chain and flexible polycarboxylate ligands: synthesis, structure, and magnetic properties. *J. Coord. Chem.* 2021, *74*, 1090–1105.
9. Xu L., Guo Y., Yuan X. *catena*-Poly[[diaquazinc(II)]-μ-4,4′-(methylenedioxy)dibenzoato]. *Acta Crystallogr.* 2009, *E65*, m360.