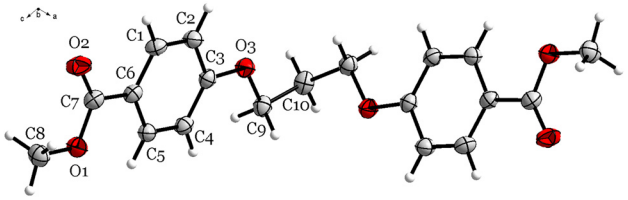
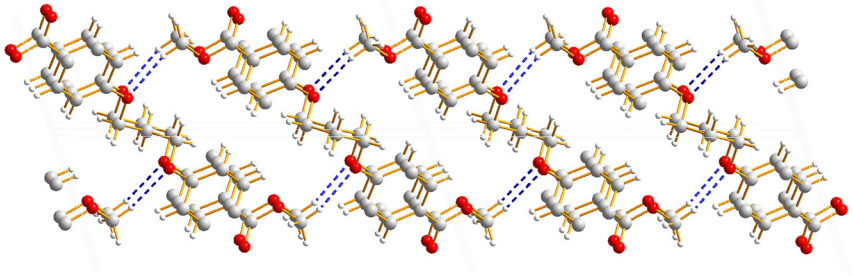
Li Miao-Miao and Zheng Yan-Ling*

Synthesis and crystal structure of dimethyl 4,4′-(propane-1,3-diylbis(oxy))dibenzoate, $C_{19}H_{20}O_6$





https://doi.org/10.1515/ncrs-2024-0102
Received March 3, 2024; accepted March 27, 2024;
published online April 5, 2024

Abstract

[$C_{19}H_{20}O_6$], monoclinic, $C2/c$ (no. 15), a = 22.467(2) Å, b = 4.9925(5) Å, c = 15.8059(17) Å, β = 108.500(1)°, V = 1681.3(3) Å^3, Z = 4, $R_{gt}(F)$ = 0.0309, w$R_{ref}(F^2)$ = 0.0852, T = 296(2) K.

CCDC no.: 2343334

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:	0.20 × 0.14 × 0.11 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.10 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	25.5°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6087, 1556, 0.019
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 1337
$N(param)_{refined}$:	116
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

1 Source of materials

Dissolve methyl 4-hydroxybenzoate (4.56 g, 0.03 mol) in acetonitrile (50 mL), add potassium carbonate (8.28 g, 0.06 mol), stir at 120 °C for an hour, and then add 1,3-dibromopropane (6.05 g, 0.3 mol) to react for 7 h. After the reaction was detected by thin layer chromatography(TLC), the acetonitrile solution was removed by steam distillation and extracted with ethyl acetate. Removal of ethyl acetate produced a colorless product. The product was filtered and washed with water (10 mL) three times. yield 45 % (based on methyl 4-hydroxybenzoate). The product was dissolved in methanol, filtered, and the filtrate was allowed to stand at room temperature. After one week, the crystals were obtained.

***Corresponding author: Zheng Yan-Ling**, Philosophy of Science and Technology, Nanchang Institute of Technology, Nanchang 330044, People's Republic of China, E-mail: 2265646942@qq.com
Li Miao-Miao, Teaching Quality Management and Evaluation Office, Nanchang Institute of Technology, Nanchang 330044, People's Republic of China. https://orcid.org/0009-0006-1476-3182

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.17252 (6)	1.1051 (3)	0.18463 (8)	0.0476 (3)
H1	0.2076	1.2012	0.2180	0.057*
C2	0.14566 (6)	0.9228 (3)	0.22618 (8)	0.0468 (3)
H2	0.1624	0.8962	0.2874	0.056*
C3	0.09332 (5)	0.7772 (2)	0.17685 (7)	0.0407 (3)
C4	0.06890 (5)	0.8137 (3)	0.08525 (8)	0.0432 (3)
H4	0.0345	0.7143	0.0518	0.052*
C5	0.09630 (5)	0.9998 (3)	0.04419 (8)	0.0427 (3)
H5	0.0798	1.0255	−0.0171	0.051*
C6	0.14799 (5)	1.1486 (2)	0.09290 (8)	0.0419 (3)
C7	0.17843 (6)	1.3512 (3)	0.05184 (8)	0.0453 (3)
C8	0.17555 (7)	1.5845 (3)	−0.07893 (9)	0.0576 (4)
H8A	0.1777	1.7546	−0.0498	0.086*
H8B	0.1501	1.6006	−0.1403	0.086*
H8C	0.2171	1.5285	−0.0759	0.086*
C9	0.01548 (6)	0.4489 (3)	0.17985 (8)	0.0470 (3)
H9A	0.0244	0.3335	0.1360	0.056*
H9B	−0.0196	0.5641	0.1495	0.056*
C10	0.0000	0.2833 (4)	0.2500	0.0502 (4)
H10A[a]	0.0355	0.1687	0.2789	0.060*
H10B[a]	−0.0355	0.1687	0.2211	0.060*
O1	0.14815 (4)	1.38818 (19)	−0.03498 (6)	0.0537 (3)
O2	0.22529 (5)	1.4710 (2)	0.09164 (7)	0.0673 (3)
O3	0.06945 (4)	0.60631 (18)	0.22494 (5)	0.0498 (3)

[a]Occupancy: 0.5.

2 Experimental details

All H atoms were included in calculated positions and refined as riding atoms, with C–H = 0.90–0.97 Å with U_{iso}(H) = 1.5 U_{eq}(C) for methyl H atoms and 1.2 U_{eq}(C) for all other H atoms.

3 Comment

Recently, the design and synthesis of metal-organic frameworks (MOFs) has become one of the intense research activity, not only owing to their intriguing variety of architectures, but also because of their potential applications as catalysis, molecular sensing, nonlinear optical and fluorescent materials [5–8]. Over the past few years, aromatic polycarboxylate ligands have been extensively employed in the synthesis of MOFs because of their potential properties and intriguing structural topologies [9]. The multifunctional organic ligands play an important role in constructing MOFs with unique structures and properties [10]. Fan et al. designed and synthesized 3,3′–Dihydr-oxy-4,4′-(propane-1,3-diyldi-oxy)dibenzaldehyde ligand [11]. Förster et al. designed and synthesized a series of three- and hexa-armed benzene derivatives featuring lateral benzoic ester and benzoic acid [12]. Qi et al. and Ma et al. reported the synthesis and crystal structure of two derivativesof ethylene-4,4′-di(oxybenzoic acid) [13, 14]. Hou et al. reported the synthesis and crystal structure of diethyl 4,4′-(butane-1,4-diyldioxy)dibenzoate [15]. Deng et al. reported the synthesis and crystal structure of 3,3′-dimeth-oxy-4,4′-(propane-1,3-diyldioxy)dibenzoic acid [16]. Recently, we also reported the synthesis and crystal structure of *p*-hydroxybenzoic acid derivative methyl 3-methoxy-4-(2-methoxy-2-oxoethoxy)benzoate [17]. We report herein the synthesis and crystal structure of dimethyl 4,4′-(propane-1,3-diylbis(oxy))dibenzoate.

In the molecules of the title structure bond lengths and angles are very similar to those given in the literature [15, 16]. In the title structure, the non hydrogen atoms of *p*-hydroxyphenylic acid in the title compound are approximately planar. The dihedral angle formed by the C1–C6 phenyl plane and the carboxlate group C7–O1–O2 plane is 4.7(8)° (upper part of the Figure). The two benzene rings in the molecule are almost perpendicular to each other, and the dihedral angle between them is 89.288 (79)°. The torsion angles of C2–C3–O3–C9, C4–C3–O3–C9, C3–O3–C9–C10 and O3–C9–C10–C9^{i}(Symmetry code: i: $-x$, y, $-z$ + 1/2) are 179.8(1)°, 0.3(2)°, –179.5(1)° and 60.9(1)°, respectively. Weak intermolecular C–H···O hydrogen bonds [C8–H8B···O3^{ii}(Symmetry code: ii: x, $-y$ + 2, z − 1/2.) 2.54 Å, 148.4°] connect two adjacent molecules into one-dimensional chain (lower part of the Figure). In addition, there also exist weak face-to-face π-π stacking interactions between the phenyl rings, which contributes to the stability of the crystal structure.

Acknowledgments: X-ray data were collected at Instrumental Analysis Center Nanchang Hangkong University, Nanchang, 330063, People's Republic of China.

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: This research was supported by the University–Industry Collaborative Education Program of Ministry of Education of China (No. 230800234154819 and 202101246033), National College Students Innovation and Entrepreneurship Training Program (No. S202312795006X and S20231279503S).

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

References

1. Bruker. *Apex2, Saint and Sadabs*; Bruker AXS Inc.: Madison, Wisconsin, USA, 2009.
2. Sheldrick G. M. A short history of *Shelx*. *Acta Crystallogr.* 2008, *A64*, 112–122.

3. Sheldrick G. M. Crystal structure refinement with SHELXL. *Acta Crystallogr.* 2015, *C71*, 3–8.
4. Brandenburg K. *DIAMOND. Visual Crystal Structure Information System. Ver. 4.0*; Crystal Impact: Bonn, Germany, 2015.
5. 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.
6. Wang S. L., Hu F. L., Zhou J. Y., Zhou Y., Huang Q., Lang J. P. Rigidity versus flexibility of ligands in the assembly of entangled coordination polymers based on bi- and tetra carboxylates and N-donor ligands. *Cryst. Growth Des.* 2015, *15*, 4087–4097.
7. Lu Y. M., Lan Y. Q., Xu Y. H., Su Z. M., Li S. L., Zang H. Y., Xu G. J. Interpenetrating metal-organic frameworks formed by self-assembly of tetrahedral and octahedral building blocks. *J. Solid State Chem.* 2009, *182*, 3105–3112.
8. Xiao P. L., Gu J., Peng D. Y., Xiong W. M., Nie X. L. Synthesis and crystal structures of two Cd(II) coordination polymers assembled by 4-carboxymethoxy-3-phenylacrylic acid ligands. *J. Chem. Crystallogr.* 2023, *53*, 16–24.
9. Manna P., Tripuramallu B. K., Bommakantia 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.
10. Wang Q. W., Wang Y. N., Qi X. F., Li X. M., Liu B. Syntheses and crystal structures of two coordination polymers of cobalt and cadmium assembled by 4,4′-(ethane-1,2-diyldioxy)dibenzoic acid. *Chin. J. Struct. Chem.* 2014, *33*, 1629–1635.
11. Fan Z., Diao C. H., Guo M. J., Yu M., Chen X. 3,3′-Dihydroxy-4,4′-(propane-1,3-diyldioxy)dibenzaldehyde. *Acta Crystallogr.* 2005, *E61*, o4004–o4005.
12. Förster S., Seichter W., Weber E. Synthesis and structures of three- and hexa-armed benzene derivatives featuring lateral benzoic ester and benzoic acid functions. *Z. Naturforsch. B Chem. Sci.* 2011, *66*, 939–949.
13. Qi G., Tang L., Li D., Fu F., Wang J., Guo L. Crystal structure of ethylene-4,4′-di(oxybenzoic acid)–4,4′-bipyridine (1:1), $C_{16}H_{14}O_6{\cdot}C_{10}H_8N_2$. *Z. Kristallogr. N. Cryst. Struct.* 2007, *222*, 237–238.
14. Ma Z., Yang H. Diethyl 4,4′-(ethane-1,2-diyldioxy)dibenzoate. *Acta Crystallogr.* 2011, *E67*, o1623.
15. Hou L. M., Kan Y.H. Diethyl 4,4′-(butane-1,4-diyldioxy)dibenzoate. *Acta Crystallogr*. 2007, *E63*, o2157–o2158.
16. Deng R. X., Liu P., Zhou W. Y. 3,3′-Dimethoxy-4,4′-(propane-1,3-diyl-dioxy)dibenzoic acid. *Acta Crystallogr.* 2007, *E63*, o4193.
17. Zheng Y. L., Li M. M. Crystal structure of methyl 3-methoxy-4-(2-methoxy-2-oxoethoxy)benzoate, $C_{12}H_{14}O_6$. *Z. Kristallogr. N. Cryst. Struct.* 2024, *239*, 355–357.