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Crystal structure of dimethyl 4,4'-oxydibenzoate, $C_{16}H_{14}O_5$

<https://doi.org/10.1515/ncrs-2018-0170>

Received May 8, 2018; accepted August 3, 2018; available online August 23, 2018

Abstract

$C_{16}H_{14}O_5$, *Ab*a2 (no. 41), $a = 7.4156(5)$ Å, $b = 31.415(2)$ Å, $c = 5.9451(4)$ Å, $V = 1384.98(16)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0316$, $wR_{ref}(F^2) = 0.0924$, $T = 297(2)$ K.

CCDC no.: 1860152

The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

In a vial flask, 11 mg (0.048 mmol) of $Zn(AcO)_2 \cdot H_2O$ was dissolved in 0.5 mL of MeOH while in another vial flask 20 mg (0.048 mmol) of the compound $C_{24}H_{18}N_4O_3$ previously reported [4] was dissolved in 0.5 mL of CH_2Cl_2 . The resulting solutions were mixture in a 5 mL vial flask and 1 mL of DMSO was added. The flask was sealed and heated at 110 °C per 48 h. Finally, the mixture was cooled gradually, obtaining a big colorless single crystal.

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Table 1: Crystal collection and handling.

Crystal:	Block, colorless
Size:	0.18 × 0.16 × 0.10 mm
Wavelength:	Mo $K\alpha$ radiation ($\lambda = 0.71073$ Å)
μ :	0.103 mm ⁻¹
Diffractometer, scan mode:	Bruker AXS D8-Venture, Φ and ω -scans
θ_{max} , completeness:	28.8°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	10890, 1707, 0.0208
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 1548
$N(param)_{refined}$:	110
Programs:	Bruker programs [1], OLEX2 [2], SHELX [3]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	U_{iso}^*/U_{eq}
O1	0.500000	0.500000	0.8451(3)	0.0401(4)
O2	0.5665(3)	0.30641(5)	0.5562(3)	0.0725(6)
O3	0.4337(2)	0.33366(4)	0.2516(3)	0.0546(4)
C1	0.4965(2)	0.38021(5)	0.5510(3)	0.0338(4)
C2	0.5763(3)	0.38758(5)	0.7590(3)	0.0385(4)
H2	0.630(3)	0.3643(7)	0.831(4)	0.046
C3	0.5804(2)	0.42817(6)	0.8493(3)	0.0364(4)
H3	0.639(3)	0.4346(6)	0.987(4)	0.044
C4	0.5010(2)	0.46144(5)	0.7330(3)	0.0311(4)
C5	0.4171(2)	0.45463(6)	0.5270(3)	0.0341(4)
H5	0.363(3)	0.4772(7)	0.440(4)	0.041
C6	0.4161(2)	0.41393(5)	0.4364(3)	0.0341(4)
H6	0.359(3)	0.4083(7)	0.298(4)	0.041
C7	0.5025(3)	0.33624(6)	0.4581(3)	0.0418(5)
C8	0.4431(4)	0.29212(8)	0.1454(5)	0.0672(7)
H8A	0.413461	0.294748	-0.011125	0.101
H8B	0.359117	0.273196	0.216842	0.101
H8C	0.562998	0.280934	0.160378	0.101

FT-IR (KBr pellet, cm⁻¹): 2958 (vw), 3014 (vw), $\nu(C-H \text{ aryl})$, 2958 (vw), 2843 (vw), $\nu(C-H \text{ alkyl})$, 1720 (vs) $\nu(C=O)$, 1284 (s) $\nu(C-O)$, 1108 (s), $\delta(C-O)$, 767(s) $\delta(C-H)$. **¹H NMR** (300 MHz, Acetone-*d*₆) δ 8.07 (d, $J = 8.9$ Hz, 4H), 7.18 (d, $J = 8.9$ Hz, 4H), 3.88 (s, 6H).

Experimental details

H atoms were located in the difference Fourier map, but refined with fixed individual displacement parameters, using

a riding model with C—H distances 0.96 Å for CH₃ with U(H) values of 1.5U_{eq}(C). The other H atoms were free refined with distances in the range of 0.94(3)–0.96(3) Å (for aromatic rings with 1.2U_{eq}(C)). The Flack-Parsons parameter isn't conclusive.

Discussion

This paper forms part of our continuing study of the synthesis and structural characterization of a flexible ligand for preparation of coordination polymers [5 and references therein]. The title compound was isolated during attempts to synthesize a coordination polymer between 4,4'-oxybis[*N*-(pyridin-4-yl)benzamide] trihydrate a flexible ligand reported for us [4] and Zn(AcO)₂ · H₂O salt.

There is one half of the title molecule in the asymmetric unit (*cf.* the figure; *i* = 1 − *x*, 1 − *y*, *z*). The O1 atom lies on two fold axis. The angles between the planes defined by C1/C2/C3/C4/C5/C6 (plane 1); C4/O1/C4ⁱ (plane 2); and C1ⁱ/C2ⁱ/C3ⁱ/C4ⁱ/C5ⁱ/C6ⁱ (plane 3) are 29.75(12)° for the planes 1 & 2 and 55.15(8)° for the planes 1 & 3. The aromatic ether bond lengths and angle are 1.3827(18) Å and 122.37(19)° respectively, which is consistent with those found in similar compounds [6]. All the other distances and angles are normal. The crystal structure is stabilized by van der Waals interaction

only. Actually, a plausible reaction mechanism of the title compound is under study.

Acknowledgements: IB Thanks to Fondecyt grant 1170256 and Fondecuip Program, grant EQM130021.

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