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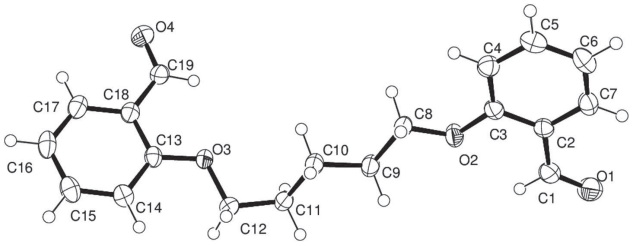
Crystal structure of 2,2′-[pentane-1,5-diylbis(oxy)] dibenzaldehyde, $C_{19}H_{20}O_4$



DOI 10.1515/ncrs-2015-0213
Received September 10, 2015; accepted February 7, 2016; available online February 26, 2016

Abstract

$C_{19}H_{20}O_4$, monoclinic, $P2_1/c$ (no. 14), $a = 16.5979(5)$ Å, $b = 8.5654(2)$ Å, $c = 11.8418(4)$ Å, $\beta = 108.537(4)°$, $V = 1596.18(8)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0385$, $wR_{ref}(F^2) = 0.0973$, $T = 190$ K.

CCDC no.: 1452071

The crystal structure is shown in the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

Salicylaldehyde (0.1 mol) and K_2CO_3 (0.1 mol) were mixed in DMF and heated to boiling temperature. 0.05 mol of 1,5-dibromopentane dissolved in DMF were then added and the reaction mixture was refluxed for 4 h. After the reaction was complete, 500 mL of water were added and the resulting percipitate was filtered and washed with water. The product was recrystallized from absolute ethanol. Single crystals suitable for diffraction experiments were obtained by slow evaporation of an ethanol solution.

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Table 1: Data collection and handling.

Crystal:	Colourless, prism, size 0.24×0.35×0.55 mm
Wavelength:	Mo K_α radiation (0.7107 Å)
μ:	0.90 cm^{-1}
Diffractometer, scan mode:	Xcalibur, Sapphire3, ω scans
$2\theta_{max}$:	53.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	11694, 3426
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3015
$N(param)_{refined}$:	208
Programs:	CrysAlis^{PRO} [11], SIR2004 [12], SHELX [13], ORTEP [14]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.5044	−0.2759	0.9176	0.043
H(4)	4*e*	0.7171	0.0536	1.1474	0.040
H(5)	4*e*	0.6750	0.1104	1.3131	0.045
H(6)	4*e*	0.5526	0.0000	1.3355	0.046
H(7)	4*e*	0.4715	−0.1706	1.1912	0.040
H(8A)	4*e*	0.7543	−0.0531	0.9909	0.038
H(8B)	4*e*	0.6827	0.0766	0.9343	0.038
H(9B)	4*e*	0.6338	−0.0770	0.7565	0.038
H(9A)	4*e*	0.7091	−0.1986	0.8128	0.038
H(10A)	4*e*	0.8103	0.0018	0.8152	0.036
H(10B)	4*e*	0.7336	0.1186	0.7533	0.036
H(11A)	4*e*	0.7744	−0.1580	0.6467	0.036
H(11B)	4*e*	0.6893	−0.0608	0.5864	0.036
H(12A)	4*e*	0.7867	0.0133	0.4935	0.037
H(12B)	4*e*	0.7757	0.1594	0.5723	0.037
H(14)	4*e*	0.8562	0.2378	0.4589	0.038
H(15)	4*e*	0.9583	0.3629	0.3944	0.043
H(16)	4*e*	1.1030	0.3248	0.4922	0.046
H(17)	4*e*	1.1458	0.1578	0.6552	0.041
H(19)	4*e*	1.0138	−0.0749	0.7751	0.038

Experimental details

All H atoms were positioned geometrically and refined using a riding model with C—H = 0.93–0.97 Å and with $U_{iso}(H) = 1.2$ times $U_{eq}(C)$.

Discussion

Aldehydes comprising two carbonyl functional groups (so called two-arm aldehydes) are promising starting materials

Table 3: Atomic coordinates and displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	4*e*	0.48905(8)	−0.2603(2)	0.9875(1)	0.0363(7)	0.0393(7)	0.0314(6)	−0.0025(6)	0.0093(5)	0.0052(5)
C(2)	4*e*	0.54368(7)	−0.1590(1)	1.0809(1)	0.0277(6)	0.0287(6)	0.0262(6)	0.0050(5)	0.0078(5)	0.0061(5)
C(3)	4*e*	0.61737(7)	−0.0909(1)	1.0679(1)	0.0286(6)	0.0289(6)	0.0257(6)	0.0069(5)	0.0097(5)	0.0043(5)
C(4)	4*e*	0.66675(8)	0.0086(2)	1.1551(1)	0.0307(6)	0.0340(7)	0.0331(6)	0.0012(5)	0.0086(5)	0.0022(5)
C(5)	4*e*	0.64160(9)	0.0414(2)	1.2537(1)	0.0450(7)	0.0348(7)	0.0293(6)	0.0014(6)	0.0068(6)	−0.0019(5)
C(6)	4*e*	0.56898(9)	−0.0240(2)	1.2676(1)	0.0504(8)	0.0375(7)	0.0308(6)	0.0078(6)	0.0193(6)	0.0022(5)
C(7)	4*e*	0.52092(8)	−0.1243(2)	1.1817(1)	0.0343(6)	0.0350(7)	0.0334(6)	0.0063(5)	0.0160(5)	0.0081(5)
C(8)	4*e*	0.69667(7)	−0.0358(2)	0.9340(1)	0.0251(6)	0.0385(7)	0.0341(6)	−0.0035(5)	0.0127(5)	−0.0012(5)
C(9)	4*e*	0.69297(8)	−0.0870(2)	0.8104(1)	0.0278(6)	0.0374(7)	0.0342(6)	−0.0041(5)	0.0145(5)	−0.0032(5)
C(10)	4*e*	0.75139(7)	0.0077(2)	0.7599(1)	0.0272(6)	0.0331(6)	0.0323(6)	−0.0028(5)	0.0128(5)	−0.0016(5)
C(11)	4*e*	0.74929(7)	−0.0520(2)	0.6376(1)	0.0240(5)	0.0360(7)	0.0301(6)	−0.0030(5)	0.0088(5)	−0.0020(5)
C(12)	4*e*	0.79639(7)	0.0505(2)	0.5759(1)	0.0240(6)	0.0389(7)	0.0285(6)	0.0010(5)	0.0074(5)	0.0032(5)
C(13)	4*e*	0.94026(7)	0.1208(1)	0.5971(1)	0.0295(6)	0.0246(6)	0.0276(6)	−0.0025(4)	0.0142(5)	−0.0037(4)
C(14)	4*e*	0.91493(8)	0.2207(2)	0.4993(1)	0.0350(6)	0.0300(6)	0.0319(6)	0.0016(5)	0.0140(5)	0.0020(5)
C(15)	4*e*	0.97581(9)	0.2951(2)	0.4613(1)	0.0495(8)	0.0268(6)	0.0377(7)	−0.0014(6)	0.0220(6)	0.0027(5)
C(16)	4*e*	1.06192(9)	0.2726(2)	0.5189(1)	0.0445(7)	0.0325(7)	0.0460(8)	−0.0117(6)	0.0264(6)	−0.0038(6)
C(17)	4*e*	1.08691(8)	0.1735(2)	0.6153(1)	0.0316(6)	0.0347(7)	0.0388(7)	−0.0077(5)	0.0159(5)	−0.0082(5)
C(18)	4*e*	1.02717(7)	0.0957(1)	0.6555(1)	0.0292(6)	0.0278(6)	0.0283(6)	−0.0036(5)	0.0128(5)	−0.0066(5)
C(19)	4*e*	1.05571(8)	−0.0162(2)	0.7546(1)	0.0261(6)	0.0405(7)	0.0292(6)	−0.0007(5)	0.0102(5)	−0.0019(5)
O(1)	4*e*	0.42585(6)	−0.3254(1)	0.99384(9)	0.0456(6)	0.0572(7)	0.0436(6)	−0.0181(5)	0.0074(5)	0.0101(5)
O(2)	4*e*	0.63525(5)	−0.1284(1)	0.96666(7)	0.0345(5)	0.0376(5)	0.0328(5)	−0.0047(4)	0.0181(4)	−0.0035(4)
O(3)	4*e*	0.88550(5)	0.0441(1)	0.64226(7)	0.0231(4)	0.0378(5)	0.0288(4)	−0.0008(3)	0.0100(3)	0.0061(4)
O(4)	4*e*	1.12945(6)	−0.0376(2)	0.81153(9)	0.0278(5)	0.0810(8)	0.0451(6)	0.0002(5)	0.0055(4)	0.0158(5)

for synthesis of macrocyclic Schiff bases. Condensation reactions of ortho substituted dialdehydes with primary amines and ketones have been recently investigated for preparation of complex macrocyclic compounds [1, 2], porous organic materials [3] and macrocyclic chalcones [4]. The title molecule is comprised of two formylphenoxy groups linked by five methylene carbon atoms (see the figure). The benzaldehyde groups enclose a dihedral angle of 67.5(6)° and therefore the molecule is non-planar. It is interesting to note, as reported in our previous contributions [5, 6], as well as in other related studies [7–9], that similar dialdehydes with even number of atoms in the aliphatic chain are planar, while molecules with odd number of atoms are non-planar. These deviations cause differences in some properties of these dialdehydes, such as melting point and crystal packing arrangement. In the crystal, the molecules are linked into dimers through weak C—H···O hydrogen bonds [graph set $R^2_2(28)$] [10]. These dimers are connected into chains along [101] direction *via* weak C—H···O interactions. Additional stabilization of the crystal structure is achieved by a number of weak C—H···O and C—H···π interactions which are linking the chains of dimers.

Acknowledgements: The authors wish to thank prof. Dubravka Matković-Čalogović for all the help during data collection and structure refinement.

References

1. Balić, T.; Marković, B.; Jaźwiński, J.; Matković-Čalogović, D.: Synthesis and structural characterization of new N_2O_2-donor Schiff base macrocycles and their silver(I) coordination polymers. Inorg. Chim. Acta **435** (2015) 283–291.
2. Ilhan, S.; Temel, H.; Yilmaz, I.; Kilic, A.: Synthesis, characterization and redox properties of macrocyclic Schiff base by reaction of 2,6-diaminopyridine and 1,3-bis(2-carboxyaldehydephenoxy)propane and its Cu(II), Ni(II), Pb(II), Co(III) and La(III) complexes. Transition Met. Chem. **32** (2007) 344–349.
3. Jin, Y., Zhu, Y.; Zhang, W.: Development of organic porous materials through Schiff-base chemistry. CrystEngComm **15** (2013) 1484–1499.
4. Mondal, R., Mandal, T. K.; Mallik, A. K.: Simple synthesis of a new family of 22- to 28-membered macrocycles containing two chalcone moieties. Arkivoc **13** (2012) 95–110.
5. Balić, T.; Marković, B.; Balić, I.: 4-[5-(4-Formylphenoxy)pentoxy] benzaldehyde. Acta Crystallogr. **E68** (2012) 2664.
6. Balić, T.; Marković, B.; Balić, I.: Triclinic polymorph of 4-[4-(4-formylphenoxy)butoxy]benzaldehyde. Acta Crystallogr. **E69** (2013) 126.
7. Ali, Q.; Raza Shah, M.; Ng, S. W.: 4,4′-(Propane-1,3-diyldioxy) dibenzaldehyde. Acta Crystallogr. **E66** (2010) 1620.
8. Dehno Khalaji, A.; Hafez Ghoran, S.; Gotoh, K.; Ishida, H.: 2-[4-(2-Formylphenoxy)butoxy]benzaldehyde. Acta Crystallogr. **E67** (2011) o2484.
9. Han, J.-R.; Zhen, X.-L.: 4-[4-(4-Formylphenoxy)butoxy] benzaldehyde. Acta Crystallogr. **E61** (2005) 4358–4359.

10. Bernstein, J.; Davis, R. E.; Shimoni, L.; Chang, N.-L.: Patterns in hydrogen bonding: functionality and graph set analysis in crystals. Angew. Chem. Int. Ed. **34** (1995) 1555–1573.
11. Oxford Diffraction: CrysAlis^{PRO} CCD and CrysAlis^{PRO} RED, including ABSPACK. Oxford Diffraction Ltd, Yarnton, England, 2009.
12. Burla, M. C.; Caliandro, R.; Camalli, M.; Carrozzini, B.; Cascarano, G. L.; DeCaro, L.; Giacovazzo, C.; Polidori, G.; Spagna, R.: SIR2004: an improved tool for crystal structure determination and refinement. J. Appl. Cryst. **38** (2005) 381–388.
13. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112–122.
14. Burnett, M. N.; Johnson, C. K.: ORTEP-III: Oak Ridge Thermal Ellipsoid Plot Program for Crystal Structure Illustrations, Oak Ridge National Laboratory Report ORNL-6895, 1996.