

1,5-Bis(2-formylphenoxy)-3-oxapentane

Nívea C. F. Dionysio, Jairo Bordinhão, Lorenzo do Canto Visentin and Célia Machado Ronconi*

Instituto de Química, Universidade Federal do Rio de Janeiro, Caixa Postal 68563, 21949-900 Rio de Janeiro, RJ, Brazil

Correspondence e-mail: ronconi@iq.ufrj.br

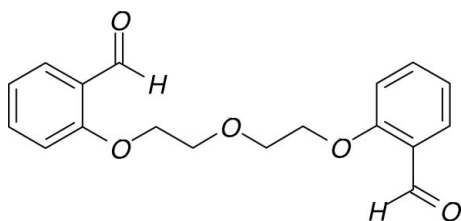
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Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.052; wR factor = 0.097; data-to-parameter ratio = 9.9.

In the title molecule, $\text{C}_{18}\text{H}_{18}\text{O}_5$, the two aromatic rings are connected by a flexible 3-oxapentane chain. The molecule has a crystallographic twofold rotation axis (C_2) passing through the central O atom. An intramolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bond is observed in the solid state.

Related literature

For related literature, see: Biernat *et al.* (1992); Qi *et al.* (2005); Jeffrey & Saenger (1991); Spek (2003).



Experimental

Crystal data

$\text{C}_{18}\text{H}_{18}\text{O}_5$

$M_r = 314.32$

Orthorhombic, $Fdd2$

$a = 27.613$ (6) Å

$b = 26.404$ (5) Å

$c = 4.4313$ (9) Å

$V = 3230.8$ (11) Å³

$Z = 8$

Mo $K\alpha$ radiation

$\mu = 0.09$ mm⁻¹

$T = 295$ (2) K

$0.25 \times 0.08 \times 0.05$ mm

Data collection

Nonius KappaCCD diffractometer

Absorption correction: none

4449 measured reflections

1043 independent reflections

480 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.134$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.051$

$wR(F^2) = 0.097$

$S = 0.99$

1043 reflections

105 parameters

H-atom parameters constrained

$\Delta\rho_{\text{max}} = 0.12$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.14$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C9}-\text{H9}\cdots\text{O2}$	0.93	2.42	2.764 (5)	102

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *PHICHI* (Duisenberg *et al.*, 2000); data reduction: *EVAL-14 (CCD)* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The X-ray diffraction measurements were performed in the Laboratório de Difração de Raios X of the Universidade Federal Fluminense (LDRX-UFF), Niterói, Brazil. The authors thank CAPES, CNPq and FAPERJ for financial support. The co-editor is thanked for the transformation of the space group $F2dd$ to the standard setting $Fdd2$, and for help with the solution and refinement of the structure.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2065).

References

- Biernat, J. F., Luboch, E., Cygan, A., Simonov, Y. A., Dvorkin, A. A., Muszalska, E. & Bilewicz, R. (1992). *Tetrahedron*, **48**, 4399–4406.
- Duisenberg, A. J. M., Hooft, R. W. W., Schreurs, A. M. M. & Kroon, J. (2000). *J. Appl. Cryst.* **33**, 893–898.
- Duisenberg, A. J. M., Kroon-Batenburg, L. M. J. & Schreurs, A. M. M. (2003). *J. Appl. Cryst.* **36**, 220–229.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Jeffrey, G. A. & Saenger, W. (1991). *Hydrogen Bonding in Biological Structures*, pp 20. Berlin: Springer-Verlag.
- Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
- Qi, A.-D., Zhu, Q.-H., He, Y.-Z. & Ren, X.-L. (2005). *Acta Cryst.* **E61**, o3784–o3785.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

supplementary materials

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Comment

The title molecule is generated by a symmetry operation according to the space group *Fdd2*. The symmetry operator (0, 1/2, *z*) applied to the equivalent atoms is a twofold rotation axis passing through the central atom O1 (Fig. 1). Bond lengths and angles are in the ranges reported for analogous structures (Biernat *et al.*, 1992; Qi *et al.*, 2005). The distances involving the C atoms in the central moiety are 1.496 (5) Å for C1—C2, 1.429 (4) Å for C1—O1, and 1.436 (4) Å for C2—O2.

The intramolecular C9—H9···O2 contact observed in the solid state (Fig. 1) is classified as a non classical hydrogen bond (Jeffrey & Saenger, 1991). This secondary interaction does not affect the torsion angles in the 3-oxapentane chain. The torsion angle for the O2/C2/C1/O1 moiety is $-69.3(3)^\circ$ and proves the flexibility of the 3-oxapentane chain. The hydrogen bonding geometry of the intramolecular contacts (Jeffrey & Saenger, 1991) with atom O2 as acceptor was calculated with *PLATON* (Spek, 2003): distances H9···O2 and C9···O2 are 2.42 and 2.764 (5) Å, respectively, and the angle at H9 is 102° .

Experimental

A mixture of 2[2-(2-*p*-tolylsulfonyloxy)ethoxy]ethanol (1 g, 8.19 mmol), salicylic aldehyde (0.87 g, 2.10 mmol), and K₂CO₃ (0.47 g, 3.40 mmol) in dry MeCN (20 ml) was heated for 12 h under reflux. After the reaction mixture had cooled down to room temperature, it was filtered and the solvent removed under vacuum. The residue was purified by column chromatography (SiO₂, Hexane:EtOAc 1:1) to give the desired product (0.55 g, 83%) as a yellow solid. A single-crystal was isolated after slow evaporation of the crystallization solvent (THF).

Refinement

All hydrogen atoms were geometrically constrained using a riding model, with C—H distances of 0.93 Å for both benzene rings and the aldehyde moieties with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}sp^2)$, and with C—H distances of 0.97 Å for the ethyl C atoms with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}sp^3)$.

Figures

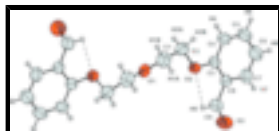


Fig. 1. *ORTEP* projection of the title molecule. Symmetry equivalent atoms are generated by the symmetry code $[-x, 1 - y, z]$. Intramolecular bonds are indicated by dashed lines. Thermal ellipsoids are shown at the 50% probability level.

1,5-Bis(2-formylphenoxy)-3-oxapentane

Crystal data

$C_{18}H_{18}O_5$	$F_{000} = 1328$
$M_r = 314.32$	$D_x = 1.292 \text{ Mg m}^{-3}$
Orthorhombic, <i>Fdd2</i>	Mo $K\alpha$ radiation
Hall symbol: F 2 -2d	$\lambda = 0.71073 \text{ \AA}$
$a = 27.613 (6) \text{ \AA}$	Cell parameters from 50 reflections
$b = 26.404 (5) \text{ \AA}$	$\theta = 1-27.5^\circ$
$c = 4.4313 (9) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$V = 3230.8 (11) \text{ \AA}^3$	$T = 295 (2) \text{ K}$
$Z = 8$	Plate, yellow
	$0.25 \times 0.08 \times 0.05 \text{ mm}$

Data collection

Nonius KappaCCD diffractometer	480 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{\text{int}} = 0.134$
Monochromator: graphite	$\theta_{\text{max}} = 27.5^\circ$
$T = 295(2) \text{ K}$	$\theta_{\text{min}} = 3.1^\circ$
φ and ω scans with κ offsets	$h = -34 \rightarrow 35$
Absorption correction: none	$k = -34 \rightarrow 34$
4449 measured reflections	$l = -5 \rightarrow 5$
1043 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.051$	H-atom parameters constrained
$wR(F^2) = 0.097$	$w = 1/[\sigma^2(F_o^2) + (0.0377P)^2]$
$S = 0.99$	where $P = (F_o^2 + 2F_c^2)/3$
1043 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
105 parameters	$\Delta\rho_{\text{max}} = 0.12 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.14 \text{ e \AA}^{-3}$
	Extinction correction: none

Special details

Experimental. The transformation of the unit cell axes and hkl intensity data were performed by using the matrix (00-1, 010, 100) in order to solve and refine the structure in the standard setting for space group *Fdd2*.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.03242 (14)	0.47183 (13)	1.1699 (9)	0.0683 (12)
H1A	0.0138	0.4508	1.3069	0.082*
H1B	0.0515	0.4951	1.2902	0.082*
C2	0.06560 (13)	0.43903 (12)	0.9883 (9)	0.0607 (10)
H2A	0.0831	0.4162	1.1207	0.073*
H2B	0.0469	0.4188	0.8472	0.073*
C3	0.13695 (13)	0.44746 (12)	0.6829 (8)	0.0495 (9)
C4	0.14652 (13)	0.39549 (12)	0.6935 (9)	0.0622 (11)
H4	0.1263	0.3740	0.8015	0.075*
C5	0.18651 (15)	0.37623 (15)	0.5414 (11)	0.0773 (13)
H5	0.1930	0.3417	0.5519	0.093*
C6	0.21687 (15)	0.40706 (15)	0.3753 (10)	0.0731 (13)
H6	0.2433	0.3935	0.2739	0.088*
C7	0.20743 (14)	0.45820 (15)	0.3617 (9)	0.0677 (13)
H7	0.2276	0.4791	0.2491	0.081*
C8	0.16770 (12)	0.47928 (12)	0.5159 (10)	0.0523 (10)
C9	0.15870 (14)	0.53391 (13)	0.4966 (13)	0.0834 (14)
H9	0.1323	0.5466	0.6030	0.100*
O1	0.0000	0.5000	0.9830 (7)	0.0529 (9)
O2	0.09910 (9)	0.47057 (7)	0.8266 (6)	0.0563 (7)
O3	0.18230 (10)	0.56356 (10)	0.3560 (9)	0.1276 (15)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.066 (3)	0.085 (3)	0.054 (3)	−0.002 (2)	−0.005 (3)	0.018 (2)
C2	0.059 (2)	0.061 (2)	0.062 (3)	−0.007 (2)	−0.011 (2)	0.023 (2)
C3	0.044 (2)	0.045 (2)	0.059 (3)	0.0026 (19)	−0.018 (2)	0.003 (2)
C4	0.060 (2)	0.044 (2)	0.083 (3)	−0.0005 (18)	−0.018 (3)	0.0096 (19)
C5	0.075 (3)	0.047 (2)	0.110 (4)	0.010 (2)	−0.029 (3)	−0.009 (3)
C6	0.069 (3)	0.066 (3)	0.084 (3)	0.013 (2)	−0.012 (3)	−0.014 (3)
C7	0.051 (3)	0.073 (3)	0.079 (3)	0.002 (2)	−0.006 (2)	0.007 (2)
C8	0.0426 (19)	0.049 (2)	0.066 (2)	0.0020 (18)	−0.014 (2)	0.011 (2)
C9	0.059 (3)	0.053 (3)	0.138 (4)	0.004 (2)	0.011 (3)	0.023 (3)
O1	0.0469 (17)	0.066 (2)	0.0457 (19)	−0.0031 (18)	0.000	0.000

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O2	0.0481 (13)	0.0462 (13)	0.0746 (18)	-0.0005 (12)	0.0020 (13)	0.0170 (14)
O3	0.090 (2)	0.0707 (18)	0.222 (4)	0.0013 (17)	0.043 (3)	0.068 (2)

Geometric parameters (\AA , $^\circ$)

C1—O1	1.429 (4)	C4—H4	0.9300
C1—C2	1.496 (5)	C5—C6	1.381 (5)
C1—H1A	0.9700	C5—H5	0.9300
C1—H1B	0.9700	C6—C7	1.377 (4)
C2—O2	1.436 (4)	C6—H6	0.9300
C2—H2A	0.9700	C7—C8	1.407 (5)
C2—H2B	0.9700	C7—H7	0.9300
C3—O2	1.368 (4)	C8—C9	1.466 (4)
C3—C4	1.398 (4)	C9—O3	1.194 (4)
C3—C8	1.405 (4)	C9—H9	0.9300
C4—C5	1.390 (5)	O1—C1 ⁱ	1.429 (4)
O1—C1—C2	111.9 (3)	C6—C5—C4	121.7 (4)
O1—C1—H1A	109.2	C6—C5—H5	119.2
C2—C1—H1A	109.2	C4—C5—H5	119.2
O1—C1—H1B	109.2	C7—C6—C5	119.1 (4)
C2—C1—H1B	109.2	C7—C6—H6	120.4
H1A—C1—H1B	107.9	C5—C6—H6	120.4
O2—C2—C1	109.1 (3)	C6—C7—C8	121.0 (4)
O2—C2—H2A	109.9	C6—C7—H7	119.5
C1—C2—H2A	109.9	C8—C7—H7	119.5
O2—C2—H2B	109.9	C3—C8—C7	119.4 (3)
C1—C2—H2B	109.9	C3—C8—C9	121.1 (4)
H2A—C2—H2B	108.3	C7—C8—C9	119.5 (4)
O2—C3—C4	124.5 (3)	O3—C9—C8	125.7 (4)
O2—C3—C8	116.1 (3)	O3—C9—H9	117.2
C4—C3—C8	119.4 (3)	C8—C9—H9	117.2
C5—C4—C3	119.5 (3)	C1 ⁱ —O1—C1	109.1 (4)
C5—C4—H4	120.2	C3—O2—C2	117.8 (2)
C3—C4—H4	120.2		
O1—C1—C2—O2	69.3 (3)	C4—C3—C8—C9	179.4 (4)
O2—C3—C4—C5	-179.1 (3)	C6—C7—C8—C3	-0.8 (6)
C8—C3—C4—C5	0.7 (5)	C6—C7—C8—C9	-180.0 (4)
C3—C4—C5—C6	-1.0 (6)	C3—C8—C9—O3	-178.5 (4)
C4—C5—C6—C7	0.5 (6)	C7—C8—C9—O3	0.6 (7)
C5—C6—C7—C8	0.4 (6)	C2—C1—O1—C1 ⁱ	176.7 (3)
O2—C3—C8—C7	-180.0 (3)	C4—C3—O2—C2	-3.0 (5)
C4—C3—C8—C7	0.2 (5)	C8—C3—O2—C2	177.2 (3)
O2—C3—C8—C9	-0.8 (5)	C1—C2—O2—C3	170.5 (3)

Symmetry codes: (i) $-x, -y+1, z$.

Hydrogen-bond geometry (\AA , $^\circ$)

D—H \cdots A	D—H	H \cdots A	D \cdots A	D—H \cdots A
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C9—H9···O2

0.93

2.42

2.764 (5)

102

Fig. 1

