organic compounds

2-[4-(2-Formylphenoxy)butoxy]-benzaldehyde

Aliakbar Dehno Khalaji,[a] Salar Hafez Ghoran,[a] Kazuma Gotoh[b] and Hiroyuki Ishida[b]*

[a]Department of Chemistry, Faculty of Science, Golestan University, Gorgan, Iran, and [b]Department of Chemistry, Faculty of Science, Okayama University, Okayama 700-8530, Japan
Correspondence e-mail: ishidah@cc.okayama-u.ac.jp

In the crystal structure of the title compound, $C_{18}H_{18}O_4$, the full molecule is generated by the application of an inversion centre. The molecule is essentially planar, with an r.m.s. deviation of 0.017 (1) Å for all non-H atoms. The molecules are linked through intermolecular C—H⋯O interactions to form a molecular sheet parallel to the ($\bar{1}$02) plane.

Related literature

For the synthesis and related structures, see: Hu *et al.* (2005); Aravindan *et al.* (2003). For related literature on Schiff bases and their transition metal complexes, see: Ilhan *et al.* (2009, 2010); Yilmaz *et al.* (2009).
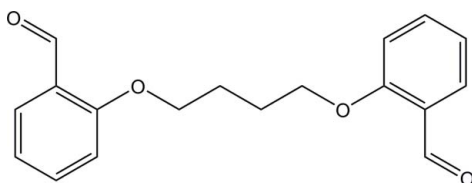
Experimental

Crystal data

$C_{18}H_{18}O_4$
$M_r = 298.34$
Monoclinic, $P2_1/c$
$a = 8.0624$ (7) Å
$b = 14.5896$ (7) Å

$c = 6.8003$ (4) Å
$\beta = 108.549$ (4)°
$V = 758.35$ (8) Å^3
$Z = 2$
Mo $K\alpha$ radiation

$\mu = 0.09$ mm^{-1}
$T = 190$ K

$0.30 \times 0.24 \times 0.15$ mm

Data collection

Rigaku R-AXIS RAPID II diffractometer
Absorption correction: numerical (*NUMABS*; Higashi, 1999)
$T_{min} = 0.980$, $T_{max} = 0.986$

12149 measured reflections
2210 independent reflections
1243 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.132$
$S = 1.13$
2210 reflections

100 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C3—H3⋯O1^i	0.95	2.53	3.397 (2)	152

Symmetry code: (i) $x + 1, -y + \frac{1}{2}, z + \frac{1}{2}$.

Data collection: *PROCESS-AUTO* (Rigaku/MSC, 2004); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure* and *PLATON* (Spek, 2009).

References

Aravindan, P. G., Yogavel, M., Thirumavalavan, M., Akilan, P., Velmurugan, D., Kandaswamy, M., Shanmuga Sundara Raj, S. & Fun, H.-K. (2003). *Acta Cryst.* E**59**, o806–o807.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Higashi, T. (1999). *NUMABS*. Rigaku Corporation, Tokyo, Japan.
Hu, P.-Z., Ma, L.-F., Wang, J.-G., Zhao, B.-T. & Wang, L.-Y. (2005). *Acta Cryst.* E**61**, o2775–o2777.
Ilhan, S., Temel, H. & Pasa, S. (2009). *Chin. Chem. Lett.* **20**, 339–343.
Ilhan, S., Temel, H., Pasa, S. & Tegin, I. (2010). *Russ. J. Coord. Chem.* **55**, 1402–1409.
Rigaku/MSC (2004). *PROCESS-AUTO* and *CrystalStructure*. Rigaku/MSC Inc., The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Yilmaz, I., Ilhan, S., Temel, H. & Kilic, A. (2009). *J. Incl. Phenom. Macrocycl. Chem.* **63**, 163–169.

supporting information

2-[4-(2-Formylphenoxy)butoxy]benzaldehyde

Aliakbar Dehno Khalaji, Salar Hafez Ghoran, Kazuma Gotoh and Hiroyuki Ishida

S1. Comment

In recent years, much attention has been paid to the synthesis and coordination chemistry of salicylaldehyde, its Schiff base derivatives and transition metal complexes (Hu *et al.*, 2005; Aravindan *et al.*, 2003; Ilhan *et al.*, 2009, 2010; Yilmaz *et al.*, 2009). The two-arm aldehydes can be condensed with primary diamines to form macrocyclic Schiff base ligands (Ilhan *et al.*, 2009, 2010; Yilmaz *et al.*, 2009).

  In the crystal structure, the title molecule, 2,2′-[butane-1,4-diylbis(oxy)]dibenzaldehyde (Fig. 1), lies on a crystallographic inversion center, thus indicating that one half the molecule comprises the asymmetric unit. The molecules are linked through intermolecular C3—H3···O1ⁱⁱ contacts (Table 1), resulting in a molecular sheet parallel to the ($\bar{1}$02) plane (Fig. 2).

S2. Experimental

The title compound was isolated from the reaction between salicylaldehyde and butane-1,4-diamine in the presence of K_2CO_3 at 85 °C for about 48 h according to the literature (Hu *et al.*, 2005). A small amount of the precipitate was dissolved in a mixture of methanol-chloroform (1:1 *v/v*) to make a clear solution and kept at room temperature for 3 days to give single crystals suitable for X-ray diffraction.

S3. Refinement

All H atoms were positioned geometrically (C—H = 0.95 or 0.99 Å) and were refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$.
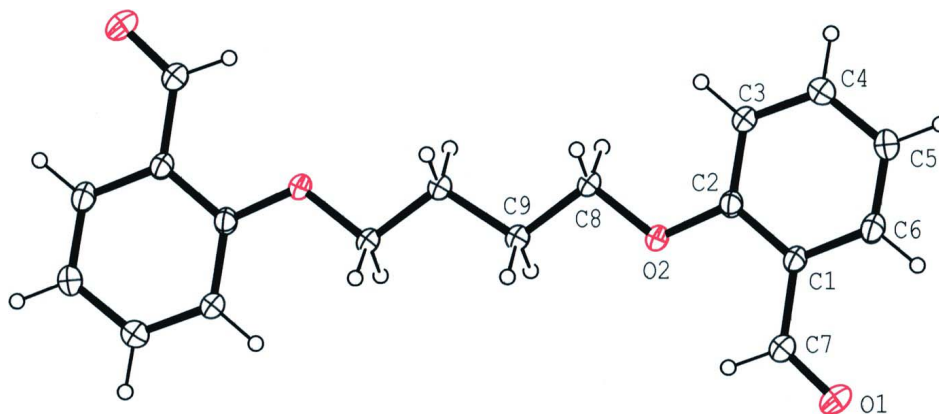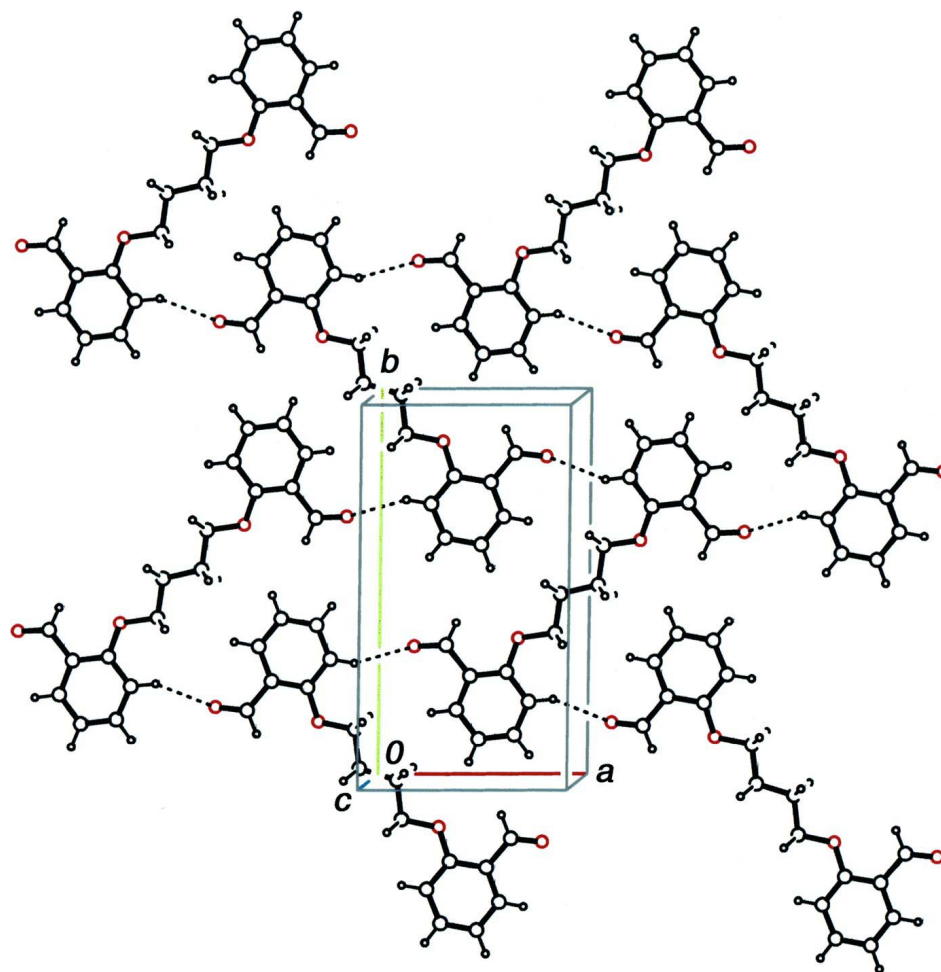

Figure 1

The molecular structure of the title compound, showing the atom-numbering scheme. Displacement ellipsoids of non-H atoms are drawn at the 50% probability level.

Figure 2

A packing diagram of the title compound, showing a molecular sheet formed by C—H···O hydrogen bonds (dashed lines).

2-[4-(2-Formylphenoxy)butoxy]benzaldehyde

Crystal data

$C_{18}H_{18}O_4$	$F(000) = 316.00$
$M_r = 298.34$	$D_x = 1.306$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71075$ Å
Hall symbol: -P 2ybc	Cell parameters from 7072 reflections
$a = 8.0624$ (7) Å	$\theta = 3.0–30.1°$
$b = 14.5896$ (7) Å	$\mu = 0.09$ mm^{-1}
$c = 6.8003$ (4) Å	$T = 190$ K
$\beta = 108.549$ (4)°	Block, pale-yellow
$V = 758.35$ (8) Å^3	$0.30 \times 0.24 \times 0.15$ mm
$Z = 2$	

Data collection

Rigaku R-AXIS RAPID II diffractometer	Absorption correction: numerical (*NUMABS*; Higashi, 1999)
Detector resolution: 10.00 pixels mm^{-1}	$T_{min} = 0.980$, $T_{max} = 0.986$
ω scans	12149 measured reflections
	2210 independent reflections

1243 reflections with $I > 2\sigma(I)$		$h = -11 \rightarrow 11$	
$R_{\text{int}} = 0.041$		$k = -20 \rightarrow 20$	
$\theta_{\text{max}} = 30.0°$		$l = -9 \rightarrow 9$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.044$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.132$	
$S = 1.13$	H-atom parameters constrained
2210 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0563P)^2 + 0.0806P]$ where $P = (F_o^2 + 2F_c^2)/3$
100 parameters	
0 restraints	$(\Delta/\sigma)_{\text{max}} = 0.0001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{max}} = 0.29$ e Å^{-3}
	$\Delta\rho_{\text{min}} = -0.24$ e Å^{-3}

Special details

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 $F^2 > \sigma(F^2)$ 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 (Å^2)

	x	y	z	$U_{\text{iso}}*/U_{\text{eq}}$
O1	0.18122 (15)	0.33844 (8)	0.1640 (2)	0.0554 (4)
O2	0.69626 (12)	0.36790 (7)	0.37956 (16)	0.0343 (3)
C1	0.45915 (17)	0.26677 (9)	0.2840 (2)	0.0307 (3)
C2	0.64045 (18)	0.27981 (9)	0.3598 (2)	0.0297 (3)
C3	0.75273 (18)	0.20492 (10)	0.4082 (2)	0.0327 (3)
H3	0.8758	0.2135	0.4596	0.039*
C4	0.6825 (2)	0.11807 (10)	0.3803 (2)	0.0380 (4)
H4	0.7586	0.0666	0.4134	0.046*
C5	0.5042 (2)	0.10385 (11)	0.3054 (3)	0.0411 (4)
H5	0.4582	0.0434	0.2868	0.049*
C6	0.3940 (2)	0.17801 (10)	0.2582 (2)	0.0380 (4)
H6	0.2712	0.1684	0.2071	0.046*
C7	0.3391 (2)	0.34439 (11)	0.2358 (3)	0.0396 (4)
H7	0.3882	0.4041	0.2625	0.047*
C8	0.88087 (17)	0.38476 (10)	0.4570 (2)	0.0333 (3)
H8A	0.9405	0.3547	0.3674	0.040*
H8B	0.9307	0.3604	0.5996	0.040*
C9	0.90444 (18)	0.48669 (10)	0.4558 (2)	0.0350 (4)
H9A	0.8379	0.5159	0.5387	0.042*
H9B	0.8568	0.5096	0.3117	0.042*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0243 (6)	0.0575 (8)	0.0782 (9)	0.0011 (5)	0.0074 (6)	−0.0063 (6)
O2	0.0210 (5)	0.0285 (5)	0.0497 (6)	−0.0040 (4)	0.0060 (4)	−0.0021 (4)
C1	0.0234 (7)	0.0324 (8)	0.0352 (7)	−0.0038 (5)	0.0077 (6)	−0.0030 (6)
C2	0.0268 (7)	0.0287 (7)	0.0333 (7)	−0.0051 (5)	0.0092 (6)	−0.0032 (6)
C3	0.0244 (7)	0.0320 (8)	0.0391 (8)	−0.0013 (6)	0.0065 (6)	−0.0020 (6)
C4	0.0358 (8)	0.0316 (8)	0.0447 (9)	0.0011 (6)	0.0102 (7)	0.0001 (7)
C5	0.0363 (8)	0.0317 (8)	0.0521 (9)	−0.0074 (6)	0.0095 (7)	−0.0039 (7)
C6	0.0286 (8)	0.0388 (9)	0.0441 (9)	−0.0085 (6)	0.0081 (7)	−0.0055 (7)
C7	0.0265 (8)	0.0372 (9)	0.0529 (10)	−0.0018 (6)	0.0095 (7)	−0.0027 (7)
C8	0.0206 (7)	0.0313 (7)	0.0453 (8)	−0.0026 (5)	0.0068 (6)	−0.0035 (6)
C9	0.0239 (7)	0.0294 (7)	0.0489 (9)	−0.0023 (6)	0.0075 (6)	−0.0035 (6)

Geometric parameters (Å, º)

O1—C7	1.2128 (18)	C5—C6	1.372 (2)
O2—C2	1.3543 (16)	C5—H5	0.9500
O2—C8	1.4333 (16)	C6—H6	0.9500
C1—C6	1.3874 (19)	C7—H7	0.9500
C1—C2	1.3997 (19)	C8—C9	1.500 (2)
C1—C7	1.458 (2)	C8—H8A	0.9900
C2—C3	1.390 (2)	C8—H8B	0.9900
C3—C4	1.376 (2)	C9—C9ⁱ	1.516 (3)
C3—H3	0.9500	C9—H9A	0.9900
C4—C5	1.379 (2)	C9—H9B	0.9900
C4—H4	0.9500		
C2—O2—C8	118.21 (11)	C5—C6—H6	119.5
C6—C1—C2	118.83 (13)	C1—C6—H6	119.5
C6—C1—C7	119.95 (13)	O1—C7—C1	124.87 (14)
C2—C1—C7	121.21 (12)	O1—C7—H7	117.6
O2—C2—C3	123.48 (13)	C1—C7—H7	117.6
O2—C2—C1	116.14 (12)	O2—C8—C9	106.66 (11)
C3—C2—C1	120.37 (12)	O2—C8—H8A	110.4
C4—C3—C2	118.85 (13)	C9—C8—H8A	110.4
C4—C3—H3	120.6	O2—C8—H8B	110.4
C2—C3—H3	120.6	C9—C8—H8B	110.4
C3—C4—C5	121.62 (14)	H8A—C8—H8B	108.6
C3—C4—H4	119.2	C8—C9—C9ⁱ	111.50 (15)
C5—C4—H4	119.2	C8—C9—H9A	109.3
C6—C5—C4	119.28 (14)	C9ⁱ—C9—H9A	109.3
C6—C5—H5	120.4	C8—C9—H9B	109.3
C4—C5—H5	120.4	C9ⁱ—C9—H9B	109.3
C5—C6—C1	121.05 (14)	H9A—C9—H9B	108.0
C8—O2—C2—C3	−0.8 (2)	C3—C4—C5—C6	−0.2 (2)

C8—O2—C2—C1	179.88 (12)	C4—C5—C6—C1	0.2 (2)
C6—C1—C2—O2	179.34 (12)	C2—C1—C6—C5	−0.1 (2)
C7—C1—C2—O2	−1.8 (2)	C7—C1—C6—C5	−179.02 (15)
C6—C1—C2—C3	0.0 (2)	C6—C1—C7—O1	−3.4 (3)
C7—C1—C2—C3	178.91 (14)	C2—C1—C7—O1	177.73 (16)
O2—C2—C3—C4	−179.30 (13)	C2—O2—C8—C9	178.18 (12)
C1—C2—C3—C4	0.0 (2)	O2—C8—C9—C9i	177.46 (15)
C2—C3—C4—C5	0.1 (2)		

Symmetry code: (i) −x+2, −y+1, −z+1.

Hydrogen-bond geometry (Å, º)

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
C3—H3⋯O1ii	0.95	2.53	3.397 (2)	152

Symmetry code: (ii) x+1, −y+1/2, z+1/2.