The 2θ scan width was $\pm(1.30 + 0.2 \tan\theta)^\circ$ in 2θ from the calculated Bragg scattering angle. Measurements were made using a scan speed of $0.04^\circ s^{-1}$ and background counts for 50% of the scan time on each side of every reflection. H atoms were positioned in geometrically idealized positions (C—H 0.97 Å). Two isotropic displacement parameters were used, one for methyl H atoms and one for phenyl and methine H atoms.

Data collection: Philips PW1100 Diffractometer Control Software. Cell refinement: Philips PW1100 Diffractometer Control Software. Data reduction: Philips PW1100 Software. Program(s) used to solve structure: *SHELX*76 (Sheldrick, 1976). Program(s) used to refine structure: *SHELX*76.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: TA1097). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). C**52**, 3234–3236

4,4′-[1,6-Hexanediylbis(oxy)]bisbenzaldehyde

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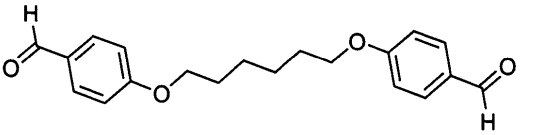
(*Received 15 May 1996; accepted 25 July 1996*)

Abstract

The title compound, $C_{20}H_{22}O_4$, shows an extended conformation for its central hexanediyl chain. The molecule is quite planar and centrosymmetric in the crystal. The structure consists of equivalent molecules stacked in layers, these layers being linked *via* electrostatic interactions between O1 of one molecule and C1 of another in the next layer.

Comment

4,4′-[1,6-Hexanediylbis(oxy)]bisbenzaldehyde, (I), is a key intermediate in the synthesis of polymer liquid crystals (Marcos, Oriol, Ros & Serrano, 1988) and dimeric liquid crystals (Date, Imrie, Luckhurst & Seddon, 1992). In the second case, there are pronounced changes in the mesomorphic properties of α,ω-bis(4-*n*-alkylaniline-benzylidine-4′-oxy)alkanes, where the parity of the flexible spacer is varied. A proposed explanation of this odd–even effect is based on the conformation of the spacer. The structure of the central hexanediyl chain of compound (I) should provide an insight into the effect of such conformations.



(I)

A perspective view of the title molecule, (I), showing the atom-numbering scheme is shown in Fig. 1. The molecular dimensions are as expected (Table 2), with aromatic C—C bond lengths ranging from 1.375 (3) to 1.389 (3) Å and aliphatic C—C bond lengths ranging from 1.503 (3) to 1.518 (3) Å. The molecule is centrosymmetric around the middle of the C10—C10($1-x, 1-y, 1-z$) bond. All non-H atoms are nearly coplanar (to within 0.2 Å), and the central hexanediyl chain shows an extended conformation.

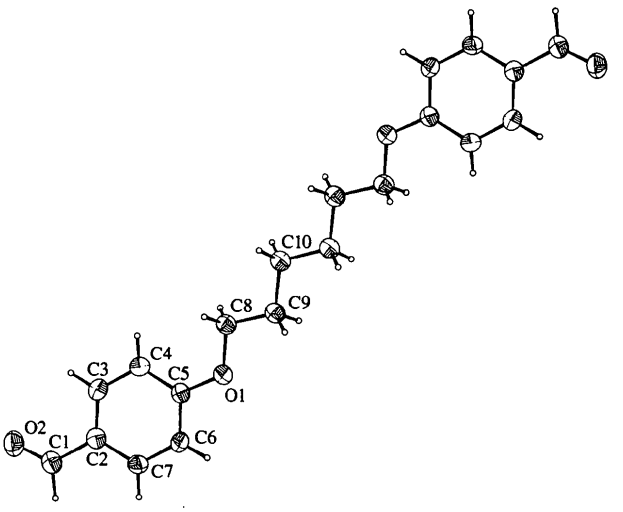


Fig. 1. Molecular structure of the title compound, showing the atomic numbering scheme. Displacement ellipsoids of the non-H atoms are drawn at the 50% probability level and H atoms are represented as spheres of arbitrary radii.

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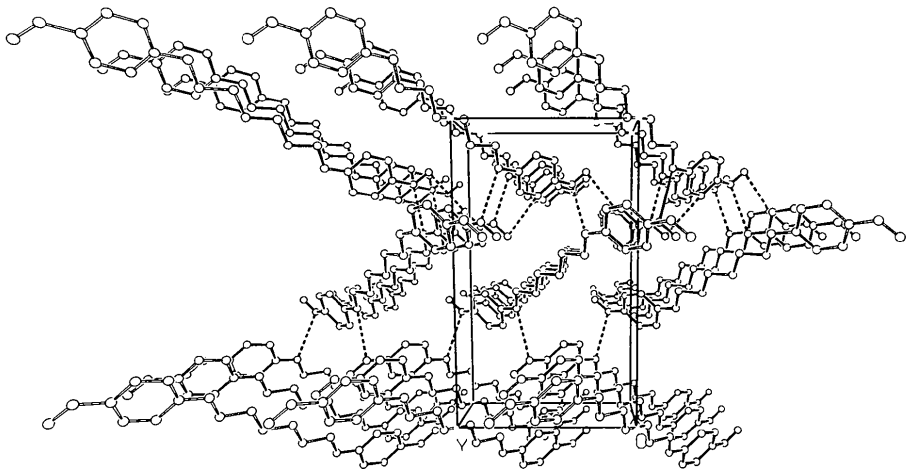

Fig. 2. The crystal packing, viewed down the x axis, showing the shortest non-bonded intermolecular distances between atoms.

The crystal packing is shown Fig. 2; molecules are stacked in layers parallel to the xy plane. The shortest non-bonded intermolecular distances between atoms are O1···C1($-\frac{1}{2} - x$, $\frac{1}{2} + y$, $\frac{3}{2} - z$) = 3.263 (3) Å and O2···C7($-\frac{3}{2} - x$, $-\frac{1}{2} + y$, $\frac{3}{2} - z$) = 3.464 (3) Å, and these close contacts are located in the interlayer planes. The interaction from O1 atoms to C1 atoms appears to be electrostatic in nature. Indeed, semi-empirical calculations using *AM*1 (Dewar, Zoebisch, Healy & Stewart, 1985) indicate that atomic charges are −0.21 e for O1 and +0.23 e for C1. The packing is different from that in the analogous compound 4,4′-[1,5-pentanediylbis-(oxy)]bisbenzonitrile (Ravikumar, Chandra Mohan, Roy & Singh, 1996) where the phenyl rings tend to stack with each other.

Experimental

The title compound was synthesized by the reaction of 1,6-dibromohexane and 4-hydroxybenzaldehyde in K_2CO_3 and DMF at 353 K over a period of 10 h (Strzelecka, Jallabert, Veber & Malthête, 1988). The product was precipitated by addition of ice and recrystallized from dichloromethane/methanol solution by slow evaporation.

Crystal data

$C_{20}H_{22}O_4$
M_r = 326.69
Monoclinic
$P2_1/n$
a = 5.268 (5) Å
b = 9.826 (7) Å
c = 16.572 (8) Å
β = 96.48 (5)°
V = 852.3 (10) Å^3
Z = 2
D_x = 1.27 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 12–14°
μ = 0.082 mm^{-1}
T = 293 K
Parallelepiped
0.5 × 0.4 × 0.2 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1640 measured reflections
1489 independent reflections
1014 observed reflections [$I > 3\sigma(I)$]

R_{int} = 0.008
θ_{max} = 25°
$h = -6 \rightarrow 6$
$k = 0 \rightarrow 11$
$l = 0 \rightarrow 19$
3 standard reflections
frequency: 120 min
intensity decay: <1%

Refinement

Refinement on F
R = 0.032
wR = 0.030
S = 2.92
1014 reflections
144 parameters
All H-atom parameters refined
Unit weights applied
$(\Delta/\sigma)_{max}$ = 0.066

$\Delta\rho_{max}$ = 0.86 e Å^{-3}
$\Delta\rho_{min}$ = −0.98 e Å^{-3}
Extinction correction: Larson (1970)
Extinction coefficient: 126
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
O1	−0.0114 (2)	0.2830 (1)	0.63932 (8)	0.0483
O2	−0.7823 (3)	−0.2206 (2)	0.6306 (1)	0.0684
C1	−0.7228 (4)	−0.1290 (2)	0.6774 (1)	0.0524
C2	−0.5273 (4)	−0.0270 (2)	0.6677 (1)	0.0420
C3	−0.3884 (4)	−0.0285 (2)	0.6016 (1)	0.0479
C4	−0.2139 (4)	0.0723 (2)	0.5899 (1)	0.0458
C5	−0.1728 (3)	0.1765 (2)	0.6461 (1)	0.0409
C6	−0.3044 (4)	0.1771 (2)	0.7141 (1)	0.0470
C7	−0.4797 (4)	0.0767 (2)	0.7243 (1)	0.0474
C8	0.1252 (4)	0.2889 (2)	0.5688 (1)	0.0443
C9	0.2806 (4)	0.4175 (2)	0.5734 (1)	0.0458
C10	0.4209 (4)	0.4356 (2)	0.4990 (1)	0.0465

Table 2. *Selected geometric parameters (Å, °)*

O1—C5	1.361 (2)	C4—C5	1.384 (3)
O1—C8	1.442 (2)	C5—C6	1.389 (3)
O2—C1	1.207 (2)	C6—C7	1.375 (3)
C1—C2	1.459 (3)	C8—C9	1.503 (3)
C2—C3	1.385 (3)	C9—C10	1.518 (3)
C2—C7	1.389 (3)	C10—C10^{i}	1.514 (4)
C3—C4	1.380 (3)		
C5—O1—C8	118.4 (1)	O1—C5—C6	116.3 (2)
O2—C1—C2	125.5 (2)	C4—C5—C6	119.2 (2)
C1—C2—C3	121.8 (2)	C5—C6—C7	120.4 (2)
C1—C2—C7	120.4 (2)	C2—C7—C6	121.0 (2)
C3—C2—C7	117.8 (2)	O1—C8—C9	108.6 (2)
C2—C3—C4	121.9 (2)	C8—C9—C10	112.0 (2)
C3—C4—C5	119.6 (2)	C9—C10—C10^{i}	113.6 (2)
O1—C5—C4	124.6 (2)		

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

The unit weights applied in the refinement gave reasonably constant average values of $<w\Delta^2>$ between different F_o and $\sin^2\theta$ intervals.

Data collection: *CAD-4/PC Software* (Enraf–Nonius, 1992). Cell refinement: *SET4* and *CELDIM* (*CAD-4/PC Software*). Data reduction: *CRYSTALS* (Watkin, Carruthers & Betteridge, 1985). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *CRYSTALS*. Molecular graphics: *CRYSTALS*. Software used to prepare material for publication: *CRYSTALS*.

This work was supported by the Centre National de la Recherche Scientifique.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1393). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). C**52**, 3236–3238

5-(2-Aminophenyl)-1,3,4-oxadiazol-2(3*H*)-one

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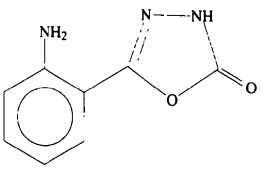
(*Received 27 August 1996; accepted 24 September 1996*)

Abstract

The title molecule, $C_8H_7N_3O_2$, is nearly planar [angle of 2.0 (1)° between the two ring planes], and contains an intramolecular N—H···N hydrogen bond and two intermolecular N—H···O hydrogen bonds

Comment

The structure of the title compound, (I), was determined to confirm the molecular structure assigned on the basis of the methods of preparation (Davidson, 1984, 1988)



(I)

and spectroscopic evidence (Tihanyi, Gál & Dvortsák, 1983). The molecule is nearly planar [with a maximum deviation of 0.037 (2) Å for O1]. The two rings are each almost exactly planar [maximum deviation of 0.012 (3) Å for C1 in the five-membered ring, and 0.004 (3) Å for three atoms in the six-membered ring], with an angle of 2.0° between the mean planes of the rings. The planarity is probably assisted by an

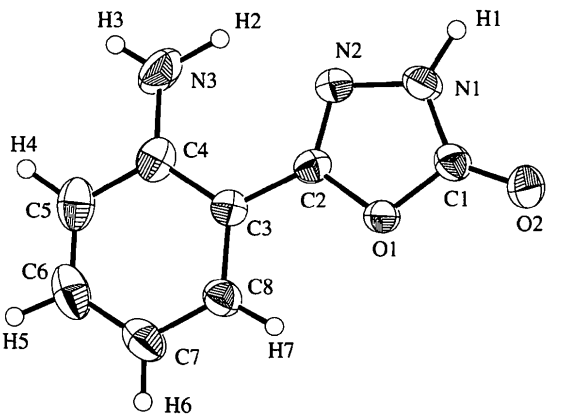


Fig. 1. View of the title molecule with displacement ellipsoids plotted at the 33% probability level. H atoms are drawn as circles of arbitrary radius.

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