P2—O12—C42	122.2 (2)	C36—C41—C40	120.0 (4)
P3—O13—C63	118.0 (2)	O12—C42—C43	110.0 (2)
P3—O15—C56	123.7 (2)	C42—C43—C44	124.3 (4)
O2—C2—C3	109.2 (3)	C42—C43—C48	117.1 (3)
O2—C2—C1	109.7 (2)	C44—C43—C48	118.6 (3)
C3—C2—C1	107.6 (2)	C43—C44—C45	120.6 (4)
O3—C3—C2	110.7 (2)	C44—C45—C46	119.8 (4)
O3—C3—C4	107.1 (3)	C45—C46—C47	119.9 (4)
C2—C3—C4	110.2 (2)	C46—C47—C48	120.7 (5)
O4—C4—C3	108.1 (2)	C43—C48—C47	120.3 (4)
O4—C4—C5	111.6 (2)	O6—C49—C50	107.7 (2)
C3—C4—C5	108.6 (3)	C49—C50—C51	120.5 (4)
O5—C5—C4	106.6 (3)	C49—C50—C55	120.7 (3)
O5—C5—C6	109.5 (2)	C51—C50—C55	118.8 (3)
C4—C5—C6	112.2 (2)	C50—C51—C52	120.0 (4)
O6—C6—C5	107.6 (2)	C51—C52—C53	119.7 (4)
O6—C6—C1	109.9 (3)	C52—C53—C54	121.0 (4)
C5—C6—C1	108.8 (2)	C53—C54—C55	119.8 (5)
O1—C1—C2	109.5 (2)	C50—C55—C54	120.6 (4)
O1—C1—C6	107.2 (2)	O15—C56—C57	111.5 (4)
C2—C1—C6	111.6 (3)	C56—C57—C58	119.0 (4)
O2—C7—C8	110.7 (3)	C56—C57—C62	121.0 (4)
C7—C8—C9	123.1 (3)	C58—C57—C62	120.0 (4)
C7—C8—C13	118.2 (4)	C57—C58—C59	119.1 (5)
C9—C8—C13	118.7 (3)	C58—C59—C60	121.8 (6)
C8—C9—C10	120.8 (3)	C59—C60—C61	117.2 (6)
C9—C10—C11	119.9 (5)	C60—C61—C62	121.0 (6)
C10—C11—C12	119.8 (5)	C57—C62—C61	120.7 (5)
C11—C12—C13	120.4 (4)	O13—C63—C64	109.3 (3)
C8—C13—C12	120.3 (5)	C63—C64—C65	119.7 (3)
O8—C14—C15	112.2 (4)	C63—C64—C69	121.0 (3)
C14—C15—C16	117.2 (4)	C65—C64—C69	119.2 (3)
C14—C15—C20	125.5 (5)	C64—C65—C66	120.3 (3)
C16—C15—C20	117.3 (3)	C65—C66—C67	119.3 (4)
C15—C16—C17	120.5 (4)	C66—C67—C68	120.5 (4)
C16—C17—C18	120.6 (6)	C67—C68—C69	120.1 (4)
C17—C18—C19	121.9 (5)	C64—C69—C68	120.6 (4)
C18—C19—C20	120.5 (5)		

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *SDP* (Frenz *et al.*, 1985). Program(s) used to solve structure: *MULTAN* (Main *et al.*, 1982) (direct methods). Program(s) used to refine structure: *SDP*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *CIF VAX MolEN* (Fair, 1990).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1235). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Conformation of Crystalline 3-(4-Methoxyphenyl)-2,2,4,4-tetramethylpentane

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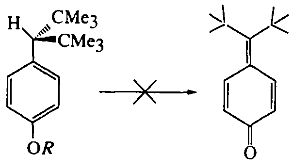
Abstract

The structure of the title compound, $C_{16}H_{26}O$, has shown that the plane of the benzene ring lies almost perpendicular [87.9 (2)°] to the plane C(2),C(3),C(4).

Comment

3-(4-Methoxyphenyl)-2,2,4,4-tetramethylpentane, (1*a*), and the corresponding phenol, (1*b*), were synthesized with the aim of converting the latter into the non-tautomerizable quinone methide, (2) (Collins & Jacobs, 1986). All attempts to oxidize (1*b*) to (2) with a variety of reagents failed, presumably because of severe non-bonded interactions between the *tert*-butyl groups and the *ortho* aromatic protons in (1*a*) (Collins & Jacobs, 1986). The quinone methide (2) remains an elusive target.



(1*a*) *R* = Me
(1*b*) *R* = H
(2)

The ^1H NMR spectra of (1*a*) and (1*b*) (Collins & Jacobs, 1986) showed asymmetric *ABCD*-type patterns for the four respective aromatic protons, indicative of

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restricted rotation at room temperature about the bond between the benzene ring and benzylic-C atom on the NMR time scale. The structure of (1*a*) has shown that the plane of the benzene ring lies almost perpendicular [87.9 (2)°] to the plane C(2),C(3),C(4) (see Fig. 1). The NMR data (Collins & Jacobs, 1986) indicate that this symmetrical 'orthogonally disposed' conformer is also the preferred species in solution.

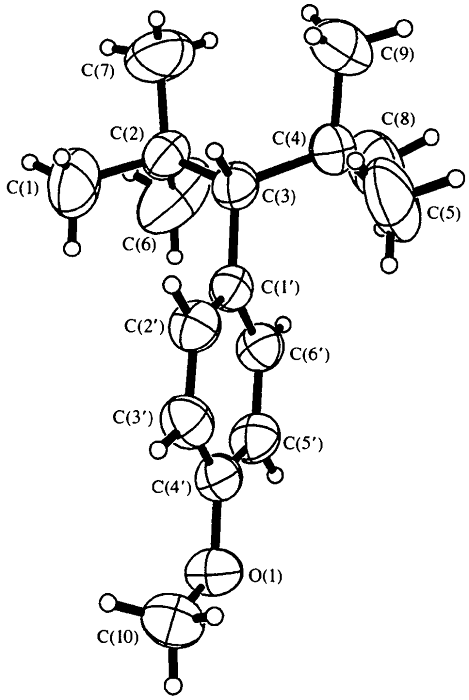

Fig. 1. Molecular structure of $C_{16}H_{26}O$ with numbering scheme. Displacement ellipsoids are scaled to 40% probability, except H atoms which, for clarity, have been reduced to spheres of arbitrary size.

Experimental

Crystals of the title compound were obtained by the method of Collins & Jacobs (1986). A representative tabular crystal was sealed in a Lindermann glass capillary along with a small amount of mother liquor to prevent decomposition.

Crystal data

$C_{16}H_{26}O$
$M_r = 234.4$
Monoclinic
$P2_1/a$
$a = 13.793\ (7)$ Å
$b = 8.247\ (4)$ Å
$c = 13.699\ (7)$ Å
$\beta = 104.52\ (1)°$
$V = 1508\ (1)$ Å^3
$Z = 4$
$D_x = 1.03$ Mg m^{-3}
$D_m = 1.01\ (2)$ Mg m^{-3}
D_m measured by flotation in CCl_4/hexane

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 24 reflections
$\theta = 6.4$–27.9°
$\mu = 0.467$ mm^{-1}
$T = 293$ K
Tabular
0.36 × 0.24 × 0.14 mm
Colourless

Data collection

PW1100 diffractometer
$\theta/2\theta$ scans
Absorption correction: face-indexed numerical (*SHELX76*; Sheldrick, 1976)
$T_{\min} = 0.893$, $T_{\max} = 0.962$
2514 measured reflections
2230 independent reflections

1189 observed reflections [$I > 3\sigma$ I]
$R_{\rm int} = 0.036$
$\theta_{\max} = 60.0°$
$h = -15 \rightarrow 14$
$k = 0 \rightarrow 9$
$l = 0 \rightarrow 15$
3 standard reflections
frequency: 240 min
intensity decay: <1%

Refinement

Refinement on F
$R = 0.066$
$wR = 0.069$
$S = 1.720$
1187 reflections
156 parameters
Only H-atom U's refined
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.166$ e Å^{-3}
$\Delta\rho_{\min} = -0.248$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from Cromer & Waber (1974) (C, O); Stewart, Davidson & Simpson (1965) (H)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

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

	x	y	z	$U_{\rm eq}$
C(1)	0.1419 (4)	0.4212 (6)	0.8194 (4)	0.136 (3)
C(2)	0.1097 (3)	0.2607 (5)	0.8599 (3)	0.073 (2)
C(3)	0.0720 (3)	0.1504 (5)	0.7653 (3)	0.065 (2)
C(4)	0.0097 (3)	−0.0067 (5)	0.7700 (3)	0.071 (2)
C(5)	−0.0069 (5)	−0.0951 (8)	0.6700 (4)	0.171 (4)
C(6)	0.1983 (4)	0.2003 (8)	0.9391 (4)	0.152 (3)
C(7)	0.0270 (4)	0.3098 (7)	0.9101 (4)	0.130 (3)
C(8)	0.0587 (4)	−0.1290 (7)	0.8500 (5)	0.135 (3)
C(9)	−0.0934 (3)	0.0293 (7)	0.7872 (5)	0.140 (4)
C(10)	0.3710 (3)	0.1390 (6)	0.4624 (3)	0.102 (3)
C(1′)	0.1538 (3)	0.1222 (5)	0.7095 (3)	0.062 (2)
C(2′)	0.1487 (3)	0.1981 (5)	0.6193 (3)	0.070 (2)
C(3′)	0.2208 (3)	0.1802 (5)	0.5657 (3)	0.073 (2)
C(4′)	0.3009 (3)	0.0823 (5)	0.6017 (3)	0.068 (2)
C(5′)	0.3091 (3)	0.0018 (5)	0.6916 (3)	0.074 (2)
C(6′)	0.2368 (3)	0.0226 (5)	0.7451 (3)	0.069 (2)
O(1)	0.3778 (2)	0.0567 (4)	0.5554 (2)	0.089 (1)

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

C(1)—C(2)	1.543 (6)	C(1′)—C(2′)	1.372 (5)
C(2)—C(3)	1.563 (5)	C(1′)—C(6′)	1.393 (5)
C(2)—C(6)	1.500 (6)	C(2′)—C(3′)	1.384 (5)
C(2)—C(7)	1.526 (5)	C(3′)—C(4′)	1.358 (5)
C(3)—C(4)	1.565 (5)	C(4′)—C(5′)	1.378 (5)
C(3)—C(1′)	1.530 (5)	C(5′)—C(6′)	1.389 (5)
C(4)—C(5)	1.518 (6)	C(4′)—O(1)	1.382 (5)
C(4)—C(8)	1.518 (6)	O(1)—C(10)	1.426 (5)
C(4)—C(9)	1.529 (6)		
C(1)—C(2)—C(3)	105.5 (3)	C(3)—C(4)—C(9)	112.8 (4)
C(1)—C(2)—C(7)	104.4 (4)	C(5)—C(4)—C(8)	105.7 (4)
C(1)—C(2)—C(6)	106.8 (4)	C(8)—C(4)—C(9)	106.3 (4)
C(3)—C(2)—C(6)	116.6 (4)	C(5)—C(4)—C(9)	107.3 (4)
C(3)—C(2)—C(7)	113.4 (3)	C(3)—C(1′)—C(2′)	120.2 (4)
C(6)—C(2)—C(7)	109.1 (4)	C(3)—C(1′)—C(6′)	123.9 (4)
C(2)—C(3)—C(4)	121.3 (3)	C(2′)—C(1′)—C(6′)	115.9 (4)
C(2)—C(3)—C(1′)	111.6 (3)	C(3′)—C(4′)—O(1)	124.9 (4)
C(4)—C(3)—C(1′)	112.1 (3)	C(5′)—C(4′)—O(1)	115.9 (4)
C(3)—C(4)—C(5)	108.7 (3)	C(4′)—O(1)—C(10)	116.8 (3)
C(3)—C(4)—C(8)	115.5 (4)		

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: *SHELX76* (Sheldrick, 1976). Program(s) used to refine structure: *SHELX76*.

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

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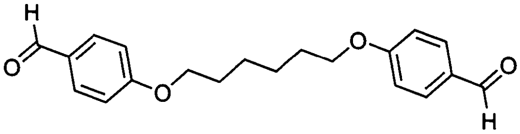
Abstract

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*-alkylanilinebenzylidine-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.

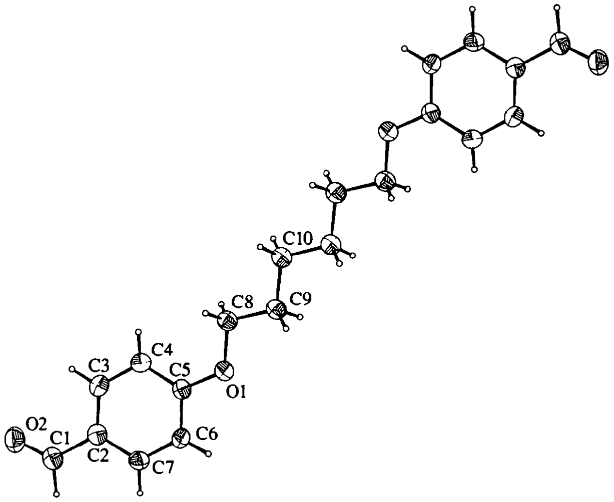


Fig. 1. Molecular structure of the title compound, showing the atomic numbering scheme. Displacement ellipsoids of the non-H atoms are drawn at the 50% probability level and H atoms are represented as spheres of arbitrary radii.

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