

2,5-Bis(methoxymethyl)-1,4-dioctyloxybenzene

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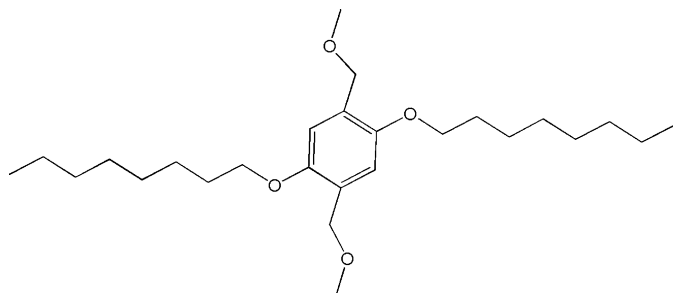
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.074; wR factor = 0.204; data-to-parameter ratio = 24.5.

The title molecule, $\text{C}_{26}\text{H}_{46}\text{O}_4$, has $\bar{1}$ symmetry. The non-H atoms of the octyloxy and methoxymethyl chains are almost coplanar with the benzene ring. There are weak $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds and $\text{C}-\text{H}\cdots\pi$ -ring interactions in the structure. The molecules are loosely packed in the structure. The unit-cell volume is 20% larger than is expected from the empirical rule pertinent to organic compounds, which states that the unit-cell volume (Å^3) is approximately equal to the number of non-H atoms in the unit cell multiplied by 18.

Related literature

For related literature regarding the synthesis of the title compound, see: Wang & Wasielewski (1997). For related literature, see: Kempster & Lipson (1972); Spek (2003).



Experimental

Crystal data

$\text{C}_{26}\text{H}_{46}\text{O}_4$
 $M_r = 422.63$

Triclinic, $P\bar{1}$
 $a = 6.6253$ (9) Å

$b = 8.8218$ (9) Å
 $c = 11.7625$ (15) Å
 $\alpha = 106.662$ (8)°
 $\beta = 95.398$ (10)°
 $\gamma = 96.066$ (9)°
 $V = 649.32$ (14) Å³

$Z = 1$
Mo $K\alpha$ radiation
 $\mu = 0.07$ mm⁻¹
 $T = 293$ (2) K
 $0.48 \times 0.38 \times 0.25$ mm

Data collection

Bruker *P4* diffractometer
Absorption correction: ψ scan
(*XSCANS*; Bruker, 1996)
 $T_{\min} = 0.746$, $T_{\max} = 0.884$
(expected range = 0.829–0.983)
4198 measured reflections

3375 independent reflections
1622 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.044$
3 standard reflections
every 97 reflections
intensity decay: 1.2%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.074$
 $wR(F^2) = 0.204$
 $S = 1.05$
3375 reflections

138 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.20$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.21$ e Å⁻³

Table 1

Geometry of hydrogen bonds and $D-\text{H}\cdots\pi$ interactions (Å, °).

Cg1 is the centroid of the $\text{C1}-\text{C3}/\text{C1}^i-\text{C3}^i$ ring.

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C1}-\text{H1A}\cdots\text{O2}^i$	0.93	2.41	2.747 (3)	101
$\text{C5}-\text{H5B}\cdots\text{Cg1}^{ii}$	0.97	2.93	3.750 (2)	143
$\text{C5}-\text{H5B}\cdots\text{Cg1}^{iii}$	0.97	2.93	3.750 (2)	143

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

Data collection: *XSCANS* (Bruker, 1996); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Bruker, 1997); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FB2057).

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supplementary materials

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Comment

The title molecule (Fig. 1) is situated on a crystallographic inversion centre. The non-hydrogen atoms of the octyloxy chains and methoxymethyl chains are almost coplanar with the benzene ring. The structure is interesting for its loose packing. The unit cell volume of the title structure is by 1.20 larger than it is expected if the 18 \AA^3 rule of the volume per non-hydrogen atom in the structure is taken into account (Kempster & Lipson, 1972). Despite the loose packing there are no large voids in the structure (Spek, 2003).

Experimental

In accordance with preparative methods of similar compounds (Wang & Wasielewski, 1997), 6 ml of the solution of HBr in acetic acid (100 ml of the latter solution contained 31 g of HBr) was added at once to a suspension of 1,4-bis(octyloxy)benzene (5.9 g, 15.1 mmol) and paraformaldehyde (0.93 g, 31.0 mmol) in acetic acid (50 ml). This mixture was then heated to 60–70 °C under stirring for 2 h.

After cooling down to room temperature, the suspension was poured into 300 ml of water. The precipitate was filtered and dissolved in 30 ml of hot chloroform (temperature: 50–55 °C), then 50 ml of methanol was added under stirring. After cooling to room temperature, the white solid was filtered off and dried under vacuum.

The white solid was dissolved in petroleum ether that boils in the interval 60–90°C. The crystals suitable for X-ray structure determination were obtained by slow evaporation at room temperature.

^1H NMR and ^{13}C NMR were determined with a Bruker Avance 400 MHz NMR spectrometer with tetramethylsilane as an internal standard.

^1H NMR (CDCl_3 , 400 MHz) δ (p.p.m.) 6.91 (s, 2 H), 4.48 (s, 4 H), 3.93 (m, 4 H), 3.42 (s, 6 H), 1.76 (m, 4 H), 1.45 (m, 4 H), 1.32 (m, 18 H), 0.89 (t, 6 H); ^{13}C NMR (CDCl_3 , 400 MHz) δ (p.p.m.) 150.0, 126.1, 111.9, 76.57, 68.73, 68.46, 57.86, 31.35, 28.98, 28.86, 28.78, 25.66, 13.60.

Refinement

All the H atoms could be found in the difference Fourier maps. Nevertheless, they were placed into the idealized positions and refined in a riding atom approximation with following constraints: $C_{\text{methyl}} - H_{\text{methyl}} = 0.96$; $C_{\text{methylene}} - H_{\text{methylene}} = 0.97$; $C_{\text{aryl}} - H_{\text{aryl}} = 0.93 \text{ \AA}$. $U_{\text{iso}}\text{H} = 1.2U_{\text{eq}}\text{C}$ except for methyls where $U_{\text{iso}}\text{H} = 1.5U_{\text{eq}}\text{C}$.

Figures

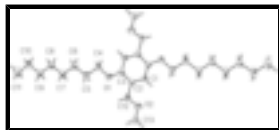


Fig. 1. The molecular structure of title compound showing 50% probability displacement ellipsoids.



Fig. 2. The packing diagram of the title structure.

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Crystal data

$C_{26}H_{46}O_4$

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Triclinic, PT

Hall symbol: $-P\ 1$

$a = 6.6253\ (9)\ \text{\AA}$

$b = 8.8218\ (9)\ \text{\AA}$

$c = 11.7625\ (15)\ \text{\AA}$

$\alpha = 106.662\ (8)^\circ$

$\beta = 95.398\ (10)^\circ$

$\gamma = 96.066\ (9)^\circ$

$V = 649.32\ (14)\ \text{\AA}^3$

$Z = 1$

$F_{000} = 234$

$D_x = 1.081\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 61 reflections

$\theta = 4.9\text{--}15.1^\circ$

$\mu = 0.07\ \text{mm}^{-1}$

$T = 293\ (2)\ \text{K}$

Prism, colourless

$0.48 \times 0.38 \times 0.25\ \text{mm}$

Data collection

Bruker P4
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293\ (2)\ \text{K}$

ω scans

Absorption correction: ψ scan
(XSCANS; Bruker, 1996)

$T_{\min} = 0.746$, $T_{\max} = 0.884$

4198 measured reflections

3375 independent reflections

1622 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.044$

$\theta_{\max} = 29.0^\circ$

$\theta_{\min} = 3.1^\circ$

$h = -8 \rightarrow 1$

$k = -11 \rightarrow 11$

$l = -16 \rightarrow 16$

3 standard reflections

every 97 reflections

intensity decay: 1.2%

Refinement

Refinement on F^2

Least-squares matrix: full

Secondary atom site location: difference Fourier map

Hydrogen site location: difference Fourier map

$$R[F^2 > 2\sigma(F^2)] = 0.074$$

$$wR(F^2) = 0.204$$

$$S = 1.05$$

3375 reflections

138 parameters

84 constraints

Primary atom site location: structure-invariant direct methods

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0902P)^2]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} < 0.001$$

$$\Delta\rho_{\max} = 0.20 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.21 \text{ e } \text{\AA}^{-3}$$

Extinction correction: none

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 $F^2 > 2\sigma(F^2)$ 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>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.3812 (2)	0.14518 (16)	0.61889 (13)	0.0605 (5)
O2	0.0976 (3)	0.33836 (18)	0.38263 (15)	0.0781 (6)
C1	0.0952 (3)	-0.0715 (2)	0.57603 (17)	0.0481 (6)
H1A	0.1603	-0.1192	0.6275	0.058*
C2	0.0963 (3)	0.1405 (2)	0.48497 (17)	0.0457 (5)
C3	0.1910 (3)	0.0668 (2)	0.56160 (18)	0.0468 (5)
C4	0.4938 (3)	0.0743 (2)	0.69460 (19)	0.0504 (6)
H4A	0.5184	-0.0312	0.6493	0.061*
H4B	0.4179	0.0642	0.7593	0.061*
C5	0.6946 (3)	0.1825 (2)	0.74413 (18)	0.0519 (6)
H5A	0.6666	0.2895	0.7830	0.062*
H5B	0.7705	0.1874	0.6784	0.062*
C6	0.8262 (3)	0.1272 (2)	0.83323 (19)	0.0515 (5)
H6A	0.7490	0.1190	0.8978	0.062*
H6B	0.8585	0.0217	0.7937	0.062*
C7	1.0233 (3)	0.2394 (2)	0.88503 (19)	0.0543 (6)
H7A	0.9905	0.3455	0.9223	0.065*
H7B	1.1014	0.2455	0.8204	0.065*
C8	1.1552 (3)	0.1891 (3)	0.97676 (19)	0.0573 (6)
H8A	1.1896	0.0838	0.9389	0.069*
H8B	1.0758	0.1810	1.0405	0.069*
C9	1.3533 (3)	0.3028 (3)	1.0317 (2)	0.0592 (6)
H9A	1.4318	0.3123	0.9679	0.071*

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H9B	1.3189	0.4078	1.0707	0.071*
C10	1.4858 (3)	0.2508 (3)	1.1217 (2)	0.0662 (7)
H10A	1.4068	0.2397	1.1849	0.079*
H10C	1.5225	0.1467	1.0824	0.079*
C11	1.6788 (4)	0.3651 (3)	1.1768 (3)	0.0897 (9)
H11A	1.7535	0.3271	1.2346	0.135*
H11B	1.6441	0.4687	1.2156	0.135*
H11C	1.7618	0.3723	1.1154	0.135*
C12	0.2062 (3)	0.2960 (2)	0.47022 (18)	0.0476 (5)
H12A	0.2199	0.3812	0.5452	0.057*
H12B	0.3424	0.2800	0.4497	0.057*
C13	0.1897 (4)	0.4850 (3)	0.3717 (2)	0.0841 (9)
H13A	0.1130	0.5101	0.3077	0.126*
H13B	0.3276	0.4759	0.3548	0.126*
H13C	0.1909	0.5683	0.4452	0.126*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0485 (9)	0.0573 (9)	0.0761 (10)	-0.0090 (7)	-0.0201 (8)	0.0353 (8)
O2	0.0733 (11)	0.0697 (10)	0.0955 (12)	-0.0189 (9)	-0.0216 (10)	0.0518 (9)
C1	0.0416 (12)	0.0495 (11)	0.0541 (12)	-0.0006 (9)	-0.0102 (10)	0.0243 (10)
C2	0.0409 (11)	0.0458 (11)	0.0502 (12)	-0.0006 (9)	-0.0039 (10)	0.0195 (9)
C3	0.0346 (11)	0.0486 (11)	0.0532 (12)	-0.0057 (9)	-0.0096 (9)	0.0177 (9)
C4	0.0410 (12)	0.0537 (12)	0.0577 (13)	-0.0012 (9)	-0.0099 (10)	0.0263 (10)
C5	0.0434 (12)	0.0516 (12)	0.0588 (13)	-0.0041 (10)	-0.0082 (10)	0.0220 (10)
C6	0.0423 (12)	0.0560 (12)	0.0553 (12)	-0.0022 (10)	-0.0083 (10)	0.0233 (10)
C7	0.0460 (13)	0.0540 (12)	0.0604 (13)	-0.0052 (10)	-0.0083 (11)	0.0222 (10)
C8	0.0444 (12)	0.0618 (13)	0.0635 (14)	-0.0050 (11)	-0.0105 (11)	0.0251 (11)
C9	0.0470 (13)	0.0623 (14)	0.0631 (14)	-0.0064 (11)	-0.0122 (11)	0.0216 (11)
C10	0.0531 (14)	0.0697 (15)	0.0700 (15)	-0.0027 (12)	-0.0175 (12)	0.0237 (12)
C11	0.0639 (17)	0.101 (2)	0.0935 (19)	-0.0119 (15)	-0.0290 (15)	0.0328 (16)
C12	0.0333 (11)	0.0479 (11)	0.0563 (12)	-0.0026 (9)	-0.0104 (9)	0.0153 (9)
C13	0.0745 (19)	0.0752 (17)	0.114 (2)	-0.0097 (14)	-0.0009 (17)	0.0585 (16)

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

O1—C3	1.382 (2)	C7—H7A	0.9700
O1—C4	1.426 (2)	C7—H7B	0.9700
O2—C12	1.363 (2)	C8—C9	1.528 (3)
O2—C13	1.416 (2)	C8—H8A	0.9700
C1—C3	1.377 (3)	C8—H8B	0.9700
C1—C2 ⁱ	1.382 (2)	C9—C10	1.514 (3)
C1—H1A	0.9300	C9—H9A	0.9700
C2—C3	1.394 (3)	C9—H9B	0.9700
C2—C12	1.547 (3)	C10—C11	1.506 (3)
C3—C2	1.394 (3)	C10—H10A	0.9700
C4—C5	1.512 (2)	C10—H10C	0.9700

C4—H4A	0.9700	C11—H11A	0.9600
C4—H4B	0.9700	C11—H11B	0.9600
C5—C6	1.519 (3)	C11—H11C	0.9600
C5—H5A	0.9700	C12—C2	1.547 (3)
C5—H5B	0.9700	C12—H12A	0.9700
C6—C7	1.513 (2)	C12—H12B	0.9700
C6—H6A	0.9700	C13—H13A	0.9600
C6—H6B	0.9700	C13—H13B	0.9600
C7—C8	1.518 (3)	C13—H13C	0.9600
C3—O1—C4	118.11 (15)	C7—C8—H8A	108.7
C12—O2—C13	111.18 (17)	C9—C8—H8A	108.7
C3—C1—C2 ⁱ	120.90 (18)	C7—C8—H8B	108.7
C3—C1—H1A	119.6	C9—C8—H8B	108.7
C2 ⁱ —C1—H1A	119.6	H8A—C8—H8B	107.6
C1 ⁱ —C2—C3	118.55 (17)	C10—C9—C8	114.05 (19)
C1 ⁱ —C2—C12	121.69 (17)	C10—C9—H9A	108.7
C3—C2—C12	119.77 (17)	C8—C9—H9A	108.7
C1—C3—O1	125.25 (18)	C10—C9—H9B	108.7
C1—C3—C2	120.56 (17)	C8—C9—H9B	108.7
O1—C3—C2	114.19 (17)	H9A—C9—H9B	107.6
O1—C4—C5	107.17 (16)	C11—C10—C9	113.6 (2)
O1—C4—H4A	110.3	C11—C10—H10A	108.8
C5—C4—H4A	110.3	C9—C10—H10A	108.8
O1—C4—H4B	110.3	C11—C10—H10C	108.8
C5—C4—H4B	110.3	C9—C10—H10C	108.8
H4A—C4—H4B	108.5	H10A—C10—H10C	107.7
C4—C5—C6	113.12 (17)	C10—C11—H11A	109.5
C4—C5—H5A	109.0	C10—C11—H11B	109.5
C6—C5—H5A	109.0	H11A—C11—H11B	109.5
C4—C5—H5B	109.0	C10—C11—H11C	109.5
C6—C5—H5B	109.0	H11A—C11—H11C	109.5
H5A—C5—H5B	107.8	H11B—C11—H11C	109.5
C7—C6—C5	112.50 (17)	O2—C12—C2	110.14 (15)
C7—C6—H6A	109.1	O2—C12—H12A	109.6
C5—C6—H6A	109.1	C2—C12—H12A	109.6
C7—C6—H6B	109.1	O2—C12—H12B	109.6
C5—C6—H6B	109.1	C2—C12—H12B	109.6
H6A—C6—H6B	107.8	H12A—C12—H12B	108.1
C6—C7—C8	113.56 (17)	O2—C13—H13A	109.5
C6—C7—H7A	108.9	O2—C13—H13B	109.5
C8—C7—H7A	108.9	H13A—C13—H13B	109.5
C6—C7—H7B	108.9	O2—C13—H13C	109.5
C8—C7—H7B	108.9	H13A—C13—H13C	109.5
H7A—C7—H7B	107.7	H13B—C13—H13C	109.5
C7—C8—C9	114.26 (18)		

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

supplementary materials

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C1—H1A \cdots O2 ⁱ	0.93	2.41	2.747 (3)	101
C5—H5B \cdots Cg1 ⁱⁱ	0.97	2.93	3.750 (2)	143
C5—H5B \cdots Cg1 ⁱⁱⁱ	0.97	2.93	3.750 (2)	143

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

Fig. 1

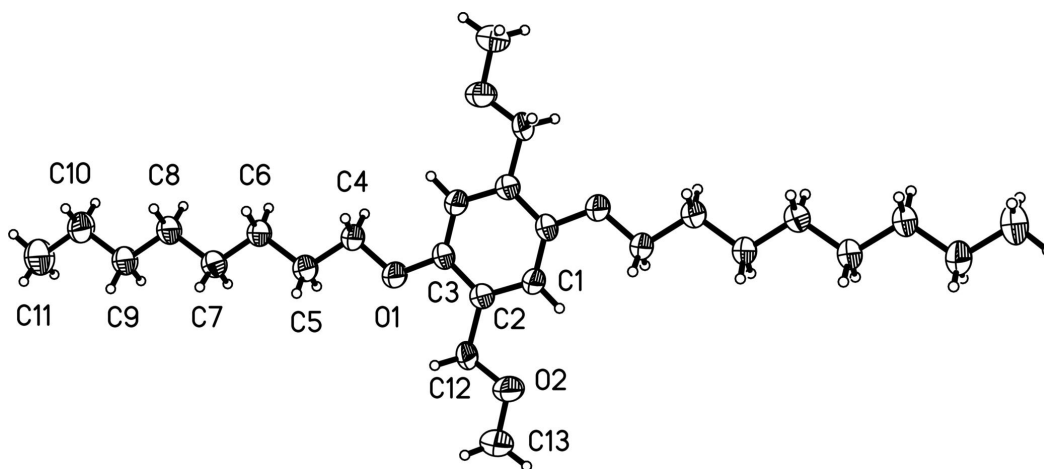


Fig. 2

