# organic papers

2,4,6-Trimethyl-1,3,5-tris(phenoxymethyl)benzene

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Key indicators

Single-crystal X-ray study
$T = 150$ K
Mean σ(C–C) = 0.003 Å
R factor = 0.034
wR factor = 0.094
Data-to-parameter ratio = 10.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The asymmetric unit of the title compound, $C_{30}H_{30}O_3$, contains two molecules with similar configurations.

Comment

We are currently investigating the supramolecular chemistry of multitopic ligands and their metal complexes. The title compound, (I), was obtained from one of a series of model experiments aimed at developing optimal conditions for the preparation of these ligands.

(I)
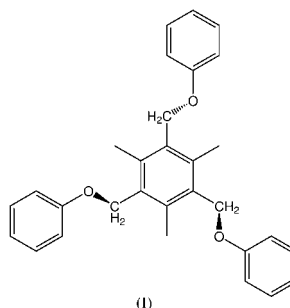
The asymmetric unit, illustrated in Fig. 1, contains two conformationally similar molecules, both in general positions. In both molecules, two of the phenoxy arms protrude on one side of the mean plane of the central benzene ring, while the other protrudes on the opposite side. The acute torsion angles that define the rotation of the H_2C-O bonds from the plane of the central benzene ring in each asymmetric molecule are listed in Table 1.

Intermolecular interactions in the crystal structure consist of edge-to-face and offset face-to-face phenyl–phenyl motifs that include the phenoxy pendant arms and the central benzene ring, although many of these display somewhat less than ideal geometric arrangements.

Experimental

1,3,5-Tris(2-bromomethyl)-2,4,6-trimethylbenzene was prepared from 1,3,5-trimethylbenzene according to the method of van der Made & van der Made (1993). Compound (I) was synthesized by a modification of a procedure for the preparation of mixed sulfur–oxygen crown macrocycles (Ashton *et al.*, 1997). Phenol (1.9 g, 20 mmol) in acetonitrile (40 ml) was added over a period of 1 h to a refluxing mixture of 1,3,5-tris(bromomethyl)-2,4,6-trimethylbenzene (2.0 g, 5.0 mmol), potassium carbonate (2.80 g, 20.3 mmol), aceto-nitrile (70 ml) and tetrahydrofuran (100 ml). The mixture was refluxed for 24 h, then filtered hot. The filtrate was evaporated under vacuum and the resulting solid was recrystallized from a mixture of acetone and water to give a fine white crystalline product (1.64 g, 75%), m.p. 440–442 K. ^{1}H NMR (CDCl$_3$, 300 MHz): δ 2.45 (9H, s, 3 ×

CH$_3$), 5.10 (6H, s, 3 × CH$_2$), 6.96–7.35 (15H, m, 3 × C$_6$H$_5$). ^{13}C NMR (CDCl$_3$, 75.5 MHz, 300 K): δ 16.28 (CH$_3$), 65.26 (CH$_2$), 115.03, 121.35, 129.91 (aromatic), 132.24, 139.70, 159.55 (quaternary aromatic). MS (ES) m/z = 461.3 $(M + Na)^+$. Elemental microanalysis, found (%): C 82.01, H 6.87; C$_{30}$H$_{30}$O$_3$ requires (%): C 82.16, H 6.89. Multi-faceted colourless crystals of sufficient quality for X-ray analysis were obtained by recrystallization from acetonitrile.

Crystal data

C$_{30}$H$_{30}$O$_3$	Mo $K\alpha$ radiation
$M_r = 438.54$	Cell parameters from 9826
Orthorhombic, $Pna2_1$	reflections
$a = 16.103$ (3) Å	$\theta = 2.3–28.2°$
$b = 10.8827$ (19) Å	$\mu = 0.08$ mm^{-1}
$c = 27.396$ (5) Å	$T = 150$ (2) K
$V = 4801.1$ (15) Å^3	Multi-faced, colourless
$Z = 8$	$0.41 \times 0.30 \times 0.25$ mm
$D_x = 1.213$ Mg m^{-3}	

Data collection

Siemens SMART 1000 CCD diffractometer	6036 independent reflections
ω scans	5477 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1999)	$R_{int} = 0.026$
	$\theta_{max} = 28.4°$
$T_{min} = 0.901$, $T_{max} = 0.980$	$h = -21 \rightarrow 21$
46 026 measured reflections	$k = -14 \rightarrow 14$
	$l = -36 \rightarrow 36$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0549P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.035$	$+ 0.8154P]$
$wR(F^2) = 0.094$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.001$
6036 reflections	$\Delta\rho_{max} = 0.24$ e Å^{-3}
601 parameters	$\Delta\rho_{min} = -0.19$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected torsion angles (°).

C2—C1—C7—O1	78.5 (2)	C36—C31—C37—O4	−80.6 (2)
C4—C3—C15—O2	−87.0 (2)	C34—C33—C45—O5	66.3 (2)
C4—C5—C23—O3	−64.7 (2)	C34—C35—C53—O6	88.1 (2)

CH H atoms were included in idealized positions and refined using a riding model, with methylene, methyl and aromatic C—H bond lengths fixed at 0.99, 0.98 and 0.95 Å, respectively. U_{iso} values were fixed at 1.2 times the U_{eq} value of the parent C atoms for methylene and aromatic H atoms and at 1.5 times the U_{eq} value of the parent C atoms for methyl H atoms. There is no crystallographic relationship between the two molecules in the asymmetric unit, confirming that the correct space group symmetry is non-centrosymmetric $Pna2_1$ and not centrosymmetric $Pnma$. Furthermore, no acceptable solution could be found in $Pnma$. The Flack (1983) parameter, x, was refined but, due to the absence of heavy atoms, a reliable value could not be obtained ($x = 0.0$, s.u 0.6). It was not, therefore, possible to assign the correct absolute structure and, accordingly, Friedel pairs were merged for the final least-squares cycles.

Data collection: SMART (Siemens, 1995); cell refinement: SAINT (Siemens, 1995); data reduction: SAINT and XPREP (Siemens, 1995); program(s) used to solve structure: SIR97 (Altomare et al., 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997) and WinGX-32
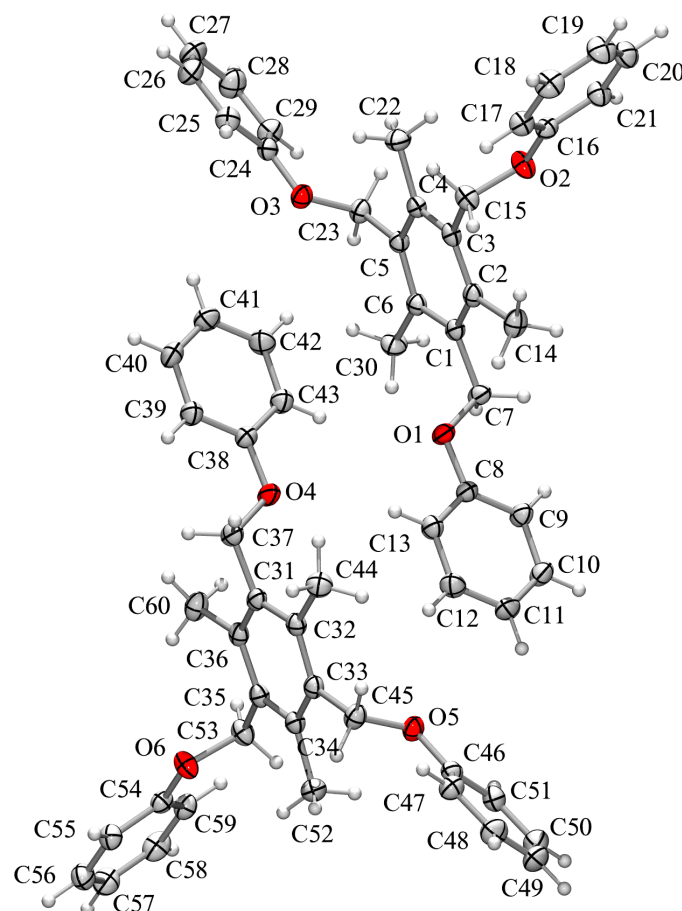
Figure 1
An ORTEP-3 plot (Farrugia, 1997) of the asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level.

(Farrugia, 1999); software used to prepare material for publication: enCIFer (CCDC, 2003).

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