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1,3-Diphenoxy-2,2-bis(phenoxymethyl)propane

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Key indicators

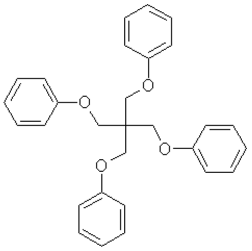
Single-crystal X-ray study
T = 220 K
Mean σ(C–C) = 0.003 Å
R factor = 0.050
wR factor = 0.141
Data-to-parameter ratio = 8.5

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

The title compound, $C_{29}H_{28}O_4$, was synthesized by a new method, crystallized from benzene/hexane, and its crystal structure determined. The molecule adopts a distorted tetrahedral geometry, and each phenyl group forms four edge-to-face phenyl–phenyl interactions with three different neighboring molecules.

Comment

Derivatives of tetraphenylmethane have been widely used as tetrahedral building blocks for molecular construction, leading to supramolecular networks, dendrimers, polymers, nanoscale structures, optoelectronic materials, liquid crystals, and other materials (Fournier, Maris *et al.*, 2003; Fournier, Wang *et al.*, 2003). The title compound, (I), provides a related subunit that is easier to make and more flexible. In the course of studying its use as a building block in supramolecular assembly, we investigated its structure to identify the preferred conformation, analyse the principal intermolecular interactions, and obtain detailed geometric information.



(I)

The title compound has been previously synthesized by the reaction of alkali salts of phenol with pentaerythrityl tetrabromide (Backer & Dijken, 1936) or with pentaerythrityl tetratosylate (Shostakovskii *et al.*, 1965). We made the compound by a modification of the second route, obtained crystals from benzene/hexane, and determined the crystal structure (Figs. 1–3). Our data confirm and significantly refine the major features of a very early structural approximation, using crystals grown from benzene/petroleum ether (Beintema *et al.*, 1935).

The two independent C2—C1—C2′ angles at the central atom C1, 108.58 (9) and 111.27 (19)°, are somewhat closer to the ideal tetrahedral value than those of tetraphenylmethane, which are approximately 107 and 111° (Robbins *et al.*, 1975). In addition, the arms connecting the central C atom to the phenyl groups are nearly fully extended, as shown by the torsion angle C1—C2—O3—C4 [175.05 (17)°]. However, the two independent C7—C1—C7′ angles defined by the central C atom and the *para* positions of the phenyl groups have the values 87.77 (5) and 121.30 (3)°, showing that, overall, the molecule deviates significantly from tetrahedral geometry.

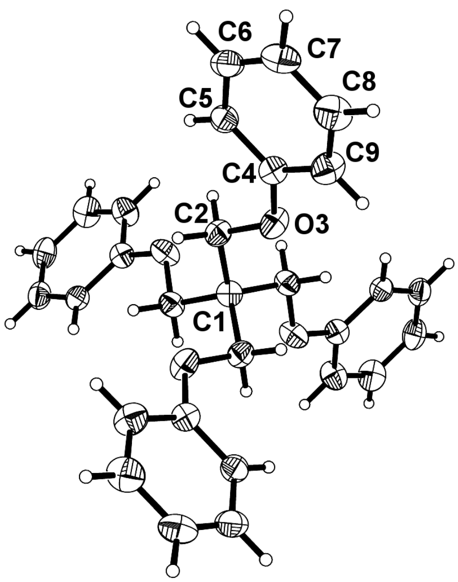


Figure 1
The structure of the title compound, with the atom-numbering scheme of the asymmetric unit. Displacement ellipsoids are drawn at the 30% probability level and H atoms are represented by spheres of arbitrary radius.

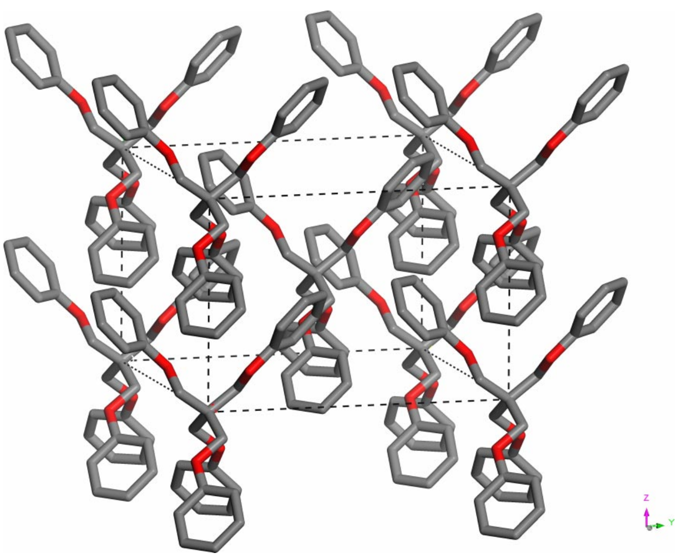


Figure 2
Packing diagram of the title compound. H atoms are omitted for clarity.

Cohesion in the crystal arises from van der Waals contacts and multiple edge-to-face phenyl–phenyl interactions. Each phenyl group participates in four of these interactions involving three neighboring molecules. Two of the four phenyl–phenyl interactions define part of a twofold embrace (Dance & Scudder, 1995), giving rise to chains along the *c* axis (Fig. 3). In these embraces, the shortest H···C distances (2.79 Å, with C—H···C angles of 150°) are between the H atom attached to C9 of one phenyl group and C6 of the other. The remaining two edge-to-face phenyl–phenyl interactions of each phenyl group involve neighbors in adjacent chains. In these interactions, the shortest H···C distances (3.16 Å, with C—H···C angles of 133°) are between the H atom attached to C6 of one phenyl group and C5 of the other.

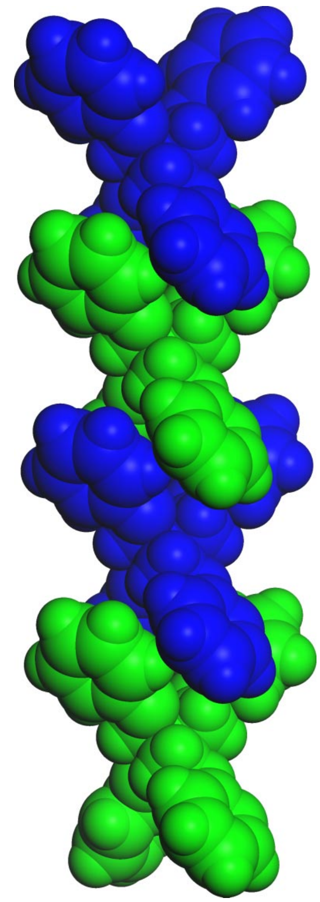


Figure 3
View of the chain parallel to the *c* axis (vertical) generated by phenyl–phenyl interactions.

Experimental

Phenol (2.50 g, 26.6 mmol) and pentaerythrityl tetratosylate (4.00 g, 5.31 mmol) were added to Cs_2CO_3 (4.33 g, 13.3 mmol) in *N,N'*-dimethylformamide (20 ml), and the mixture was heated at 413 K for 24 h. Water was then added, the resulting mixture was extracted with diethyl ether, and the organic phase was washed with water and dried over anhydrous $MgSO_4$. Evaporation of solvent under reduced pressure left a residue which was filtered over silica gel, using chloroform as eluant, and then crystallized from benzene/hexane to give crystals of the title compound (1.25 g, 2.84 mmol, 53%).

Crystal data

$C_{29}H_{28}O_4$
M_r = 440.51
Tetragonal, $I\bar{4}$
a = 12.2242 (3) Å
c = 8.4655 (3) Å
V = 1265.01 (6) Å^3
Z = 2
D_x = 1.156 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 2339 reflections
θ = 5.1–69.7°
μ = 0.61 mm^{-1}
T = 220 (2) K
Block, colorless
0.20 × 0.15 × 0.15 mm

Data collection

Bruker AXS SMART 2K/Platform diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.896, T_{max} = 0.913
3443 measured reflections
643 independent reflections
613 reflections with $I > 2\sigma(I)$
R_{int} = 0.059
θ_{max} = 70.0°
$h = -14 \rightarrow 14$
$k = -14 \rightarrow 14$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.141$
$S = 1.08$
643 reflections
76 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1128P)^2 + 0.0245P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.013 (2)

Table 1
Selected geometric parameters (Å, °).

C1—C2	1.527 (2)	C5—C6	1.382 (4)
C2—O3	1.419 (3)	C6—C7	1.373 (4)
O3—C4	1.362 (3)	C7—C8	1.392 (5)
C4—C5	1.386 (3)	C8—C9	1.371 (4)
C4—C9	1.386 (3)		
C2^{i}—C1—C2	108.58 (9)	C5—C4—C9	119.9 (2)
C2—C1—C2^{ii}	111.27 (19)	C6—C5—C4	119.2 (2)
O3—C2—C1	108.23 (15)	C7—C6—C5	121.4 (2)
C4—O3—C2	117.91 (16)	C6—C7—C8	119.0 (3)
O3—C4—C5	124.6 (2)	C9—C8—C7	120.4 (3)
O3—C4—C9	115.50 (19)	C8—C9—C4	120.2 (2)
C2^{i}—C1—C2—O3	61.53 (10)	C9—C4—C5—C6	1.6 (4)
C2^{ii}—C1—C2—O3	−57.91 (13)	C4—C5—C6—C7	−0.5 (4)
C2^{iii}—C1—C2—O3	−177.36 (18)	C5—C6—C7—C8	−0.6 (4)
C1—C2—O3—C4	175.05 (17)	C6—C7—C8—C9	0.6 (5)
C2—O3—C4—C5	11.9 (3)	C7—C8—C9—C4	0.5 (5)
C2—O3—C4—C9	−169.3 (2)	O3—C4—C9—C8	179.6 (3)
O3—C4—C5—C6	−179.7 (2)	C5—C4—C9—C8	−1.6 (4)

Symmetry codes: (i) $1 + y, -1 - x, -z$; (ii) $-x, -2 - y, z$; (iii) $-1 - y, x - 1, -z$.

As no elements heavier than oxygen are present, the Friedel pairs were merged and the absolute structure could not be determined. H atoms were placed in idealized positions with their isotropic displacement parameters fixed to $1.2U_{eq}$ of the atoms to which they are bonded.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1999); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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