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Bis[2-(2-hydroxyphenoxy)ethyl] ether methanol solvate

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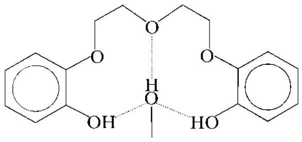
The title compound, 2,2′-(3-oxapentane-1,5-diyldioxy)diphenol, crystallizes as a methanol solvate, $C_{16}H_{18}O_5{\cdot}CH_4O$. The methanol molecule, roughly perpendicular to the mean plane of the polyether molecule, is hydrogen bonded to the two polyether OH groups and to the central ether O atom. Possible C—H$\cdots\pi$ intermolecular interactions are present.

Comment

In the course of our studies on complexes of catechol derivatives, we determined the crystal structure of bis[2-(2-hydroxyphenoxy)ethyl] ether methanol solvate, (I).



(I)

The crystal structures of only two related compounds have been reported previously. One corresponds to the molecule with two methoxy groups in place of the OH groups in (I) (Chacko *et al.*, 1984) and the other to the potassium picrate complex of the molecule with two carboxymethoxy substituents (Hughes *et al.*, 1978). In the first of these, the catechol units are located *trans* to the central ether moiety, whereas in the second, the arrangement is *cis* due to complexation to potassium ions.

The asymmetric unit of (I) comprises one bis[2-(2-hydroxyphenoxy)ethyl] ether and one methanol molecule (Fig. 1). The geometry of the former is *cis* due to the involvement of the methanol molecule in hydrogen bonding. The methanol hydroxyl H atom is bound to the central ether O atom, whereas the methanol O atom is hydrogen bonded to the two OH groups of the catechol units. The methanol O atom is thus involved in ring closure. The five atoms O1, O2, O3, O4 and O5 define a mean plane with a maximum deviation of 0.071 (1) Å, with the methanol O6 atom lying 0.708 (1) Å from this plane. The situation is somewhat different in the hydrated form of the analogous compound with an ether chain longer by one $O(CH_2)_2$ unit (Suh *et al.*, 1985), since in this case the array of O atoms is sufficiently large to encompass the water molecule close to its centre with, furthermore, one of the catechol hydroxyl H atoms involved in an intermolecular hydrogen bond.

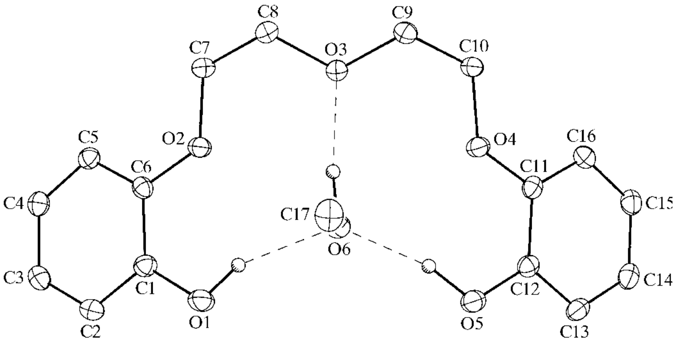


Figure 1
View of the title molecule with the atomic numbering scheme. Hydrogen bonds are shown as dashed lines and displacement ellipsoids are drawn at the 30% probability level.

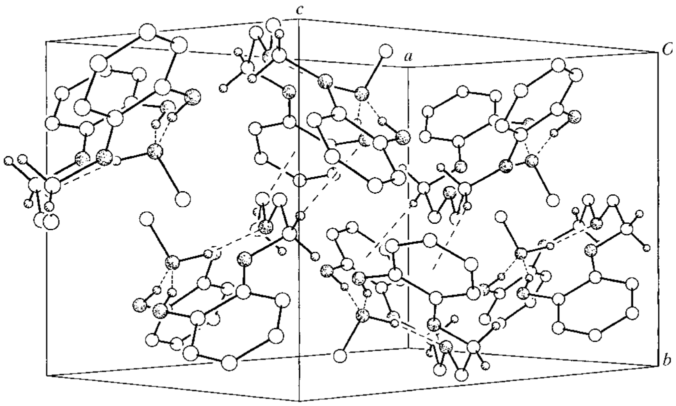


Figure 2
The packing arrangement of (I). H atoms have been omitted for clarity, except those involved in hydrogen bonding and those on C7 and C10. Hydrogen bonds are shown as dashed lines.

The dihedral angle between the two aromatic rings in (I) is 117.76 (6)°, which gives the molecule a 'butterfly' shape, the methanol molecule being located on the convex side. The O—C—C—O torsion angles in the ether part of the molecule are 61.4 (2) and −64.3 (2)° for O2—C7—C8—O3 and O3—C9—C10—O4, respectively, close to the ideal values for *gauche* angles, whereas the C—O—C—C torsion angles deviate from their ideal values of 0 or 180° by less than 10°.

The crystal packing contains an interesting feature. Molecules are related by the screw axis such that two H atoms of the ether chain (bound to C7 and C10) point towards the centroids of the aromatic rings of a neighbouring molecule. The H$\cdots$centroid distances (2.56 and 2.58 Å) and the C—H$\cdots$centroid angles (151 and 147°) indicate the possibility of weak C—H$\cdots\pi$ interactions, at least on geometrical grounds (Jeffrey & Saenger, 1994).

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Experimental

Bis[2-(2-hydroxyphenoxy)ethyl] ether was synthesized according to a previously reported procedure (Kyba *et al.*, 1977) and was recrystallized from methanol.

Crystal data

$C_{16}H_{18}O_5 \cdot CH_4O$
$M_r = 322.35$
Monoclinic, $P2_1/c$
$a = 10.8638$ (7) Å
$b = 9.6264$ (5) Å
$c = 15.9399$ (9) Å
$\beta = 94.270$ (3)°
$V = 1662.4$ (2) Å^3
$Z = 4$
$D_x = 1.288$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6010 reflections
$\theta = 2.8$–25.7°
$\mu = 0.10$ mm^{-1}
$T = 100$ (2) K
Parallelepiped, colourless
0.30 × 0.30 × 0.25 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ scans
6010 measured reflections
3129 independent reflections
2190 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.047$
$\theta_{\rm max} = 25.7°$
$h = -13 \rightarrow 13$
$k = -11 \rightarrow 11$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.119$
$S = 1.02$
3129 reflections
209 parameters
H atoms: see text
$w = 1/[\sigma^2(F_o^2) + (0.055P)^2 + 0.291P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.16$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.23$ e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O6	0.90	1.90	2.7566 (19)	158
O5—H5···O6	0.99	1.78	2.7392 (18)	163
O6—H6···O3	1.03	1.84	2.8485 (18)	167

The hydroxyl H atoms of the methanol molecule and catechol units were found in the Fourier difference map and were introduced as riding atoms with a displacement parameter equal to 1.2 times that of the parent atom. The O6—H6 bond length was slightly larger than usual, but was kept as found. All other H atoms were introduced at calculated positions (CH = 0.93, CH_2 = 0.97 and CH_3 = 0.96 Å) as riding atoms with an isotropic displacement parameter equal to 1.2 (CH and CH_2) or 1.5 (CH_3) times that of the parent atom.

Data collection: *KappaCCD Software* (Nonius, 1998); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL* and *PARST*97 (Nardelli, 1995).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1474). Services for accessing these data are described at the back of the journal.

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