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2,2′-(*p*-Phenylenedimethylene)bis(propane-1,3-diol)

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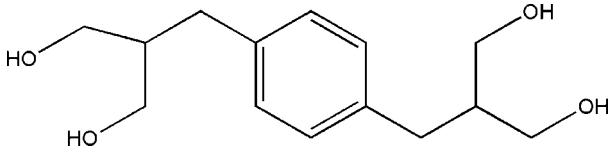
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.002 Å; R factor = 0.038; wR factor = 0.115; data-to-parameter ratio = 18.6.

The molecule of the title compound, $C_{14}H_{22}O_4$, is centrosymmetric. In the crystal, the molecules are linked through O—H···O hydrogen bonds into a three-dimensional network.

Related literature

For a related structure, see: Xi *et al.* (2008).



Experimental

Crystal data

$C_{14}H_{22}O_4$
M_r = 254.32
Orthorhombic, *Pbca*
a = 9.939 (6) Å
b = 8.803 (5) Å
c = 15.366 (9) Å
V = 1344.5 (14) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 291 (2) K
0.30 × 0.24 × 0.22 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.97, T_{max} = 0.98
7571 measured reflections
1636 independent reflections
1215 reflections with $I > 2\sigma(I)$
R_{int} = 0.057

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.115
S = 1.08
1636 reflections
88 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···O2^{i}	0.820 (16)	1.906 (17)	2.7254 (17)	177.4 (17)
O2—H2A···O1^{ii}	0.820 (17)	1.943 (17)	2.7612 (17)	175.5 (17)

Symmetry codes: (i) $x+\frac{1}{2}, y, -z+\frac{3}{2}$; (ii) $-x+2, y-\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2172).

References

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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Xi, H., Gao, Y., Sun, X., Meng, Q. & Jiang, Y. (2008). *Acta Cryst.* E**64**, o1853.

supporting information

Acta Cryst. (2009). E**65**, o170 [doi:10.1107/S1600536808041688]

2,2′-(*p*-Phenylenedimethylene)bis(propane-1,3-diol)

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S1. Comment

Reduction is a fundamental transformation in organic synthesis. Lithium aluminium hydride is used in organic synthesis as a powerful reducing agent.

The title molecule has a crystallographic inversion center located at the middle of the benzene ring. The *trans*-arrangement of two 1,3-dihydroxyisopropyl groups in the title compound was verified by X-ray crystallographic studies (Fig.1). The torsion angle C3—C4—C5—C6 is 165.14 (10) ° and the torsion angle of C3-C4-C5- C7 is -70.96 (13).

S2. Experimental

Tetraethyl 2,2′-(*p*-phenylenedimethylene)dimalonate was prepared according to the literature procedure (Xi *et al.*, 2008). In a flame-dryed, round-bottom flask was placed freshly distilled THF (80 ml) under dry nitrogen gas and the flask was placed in an ice-bath. Subsequently $LiAlH_4$ (2.128 g, 56 mmol) was slowly added with stirring, followed by a dropwise addition of the solution of tetraethyl 2,2′-(*p*-phenylenedimethylene)dimalonate (2.95 g,7 mmol) in THF (20 ml) . After stirring for 3 h at room temperature, a saturated solution of Na_2SO_4(3 ml) was added. Stirring was continued for next 10 min. Then ethanol (8 mL) was added, the mixture was heated to 333 K. Lithium and aluminium salts were separated by filtration on celite. Filtrate was evaporated and the residue purified by crystallization, yielding the title compound (1.09 g, yield 61%; m.p. 448–449 K). Crystals suitable for X-ray analysis were obtained by slow evaporation of an aqueous solution at 288 K.

S3. Refinement

Carbon bound H atoms were placed geometrically and treated as riding on their carriers, with methylene C—H distance of 0.97 Å, aromatic C—H of 0.93 Å and U_{iso}(H) = 1.2Ueq(C). H atoms from hydroxyl groups were refined with the distance restraint of O—H = 0.82 (2) Å and U_{iso}(H) = 1.5 U_{eq}(O)..

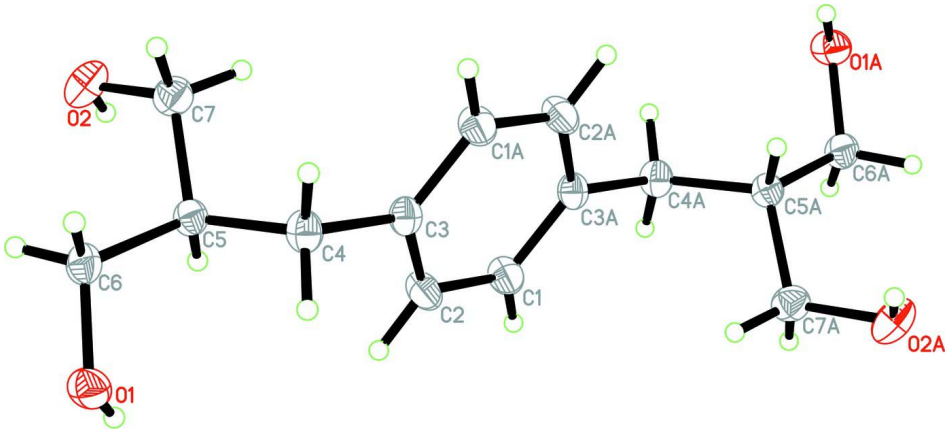

Figure 1

View of the title molecule showing the atom-labelling scheme; displacement ellipsoids are shown at the 30% probability level (symmetry code to generate atoms with the label A: 2-x, 1-y, 1-z)

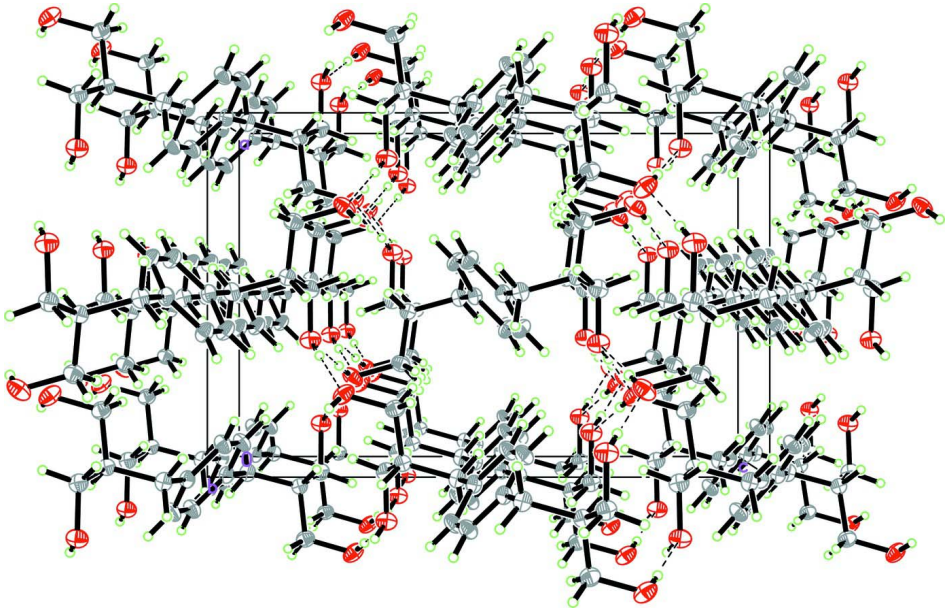

Figure 2

Crystal packing of the title compound viewed down the b direction. Dashed lines indicate hydrogen bonds.

2,2′-(*p*-Phenylenedimethylene)bis(propane-1,3-diol)

Crystal data

$C_{14}H_{22}O_4$
$M_r = 254.32$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 9.939\ (6)$ Å
$b = 8.803\ (5)$ Å
$c = 15.366\ (9)$ Å
$V = 1344.5\ (14)$ Å^3
$Z = 4$
$F(000) = 552$
$D_x = 1.256$ Mg m^{-3}
Melting point = 448–449 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2317 reflections
$\theta = 2.6$–$27.1°$
$\mu = 0.09$ mm^{-1}
$T = 291$ K
Block, colourless
0.30 × 0.24 × 0.22 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.97, T_{max} = 0.98
7571 measured reflections
1636 independent reflections
1215 reflections with $I > 2\sigma(I)$
R_{int} = 0.057
θ_{max} = 28.3°, θ_{min} = 2.7°
$h = -5 \rightarrow 13$
$k = -11 \rightarrow 11$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.115
S = 1.08
1636 reflections
88 parameters
2 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.06P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	1.09492 (13)	0.40412 (13)	0.53366 (8)	0.0388 (3)
H1	1.1600	0.3402	0.5572	0.047*
C2	1.09178 (14)	0.55517 (13)	0.55850 (8)	0.0396 (3)
H2	1.1548	0.5910	0.5982	0.048*
C3	0.99643 (11)	0.65355 (12)	0.52522 (8)	0.0316 (3)
C4	0.99230 (13)	0.81711 (12)	0.55423 (8)	0.0347 (3)
H4A	0.9288	0.8719	0.5180	0.042*
H4B	1.0804	0.8620	0.5454	0.042*
C5	0.95220 (12)	0.83713 (12)	0.64958 (7)	0.0303 (3)
H5	1.0045	0.7646	0.6842	0.036*
C6	0.98372 (12)	0.99558 (14)	0.68286 (9)	0.0371 (3)
H6A	0.9369	1.0694	0.6471	0.045*
H6B	0.9502	1.0053	0.7419	0.045*
C7	0.80474 (13)	0.80040 (14)	0.66200 (8)	0.0399 (3)
H7A	0.7512	0.8777	0.6333	0.048*

H7B	0.7853	0.7039	0.6342	0.048*
O1	1.12348 (9)	1.02918 (10)	0.68214 (6)	0.0417 (3)
H1A	1.1656 (18)	0.9562 (18)	0.7010 (10)	0.063*
O2	0.76614 (10)	0.79212 (10)	0.75078 (6)	0.0471 (3)
H2A	0.8000 (18)	0.7167 (18)	0.7731 (12)	0.071*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0425 (7)	0.0340 (7)	0.0399 (7)	0.0101 (5)	−0.0108 (5)	−0.0035 (5)
C2	0.0430 (7)	0.0361 (7)	0.0398 (7)	0.0027 (5)	−0.0128 (6)	−0.0076 (5)
C3	0.0383 (7)	0.0269 (6)	0.0297 (6)	0.0000 (5)	0.0018 (5)	−0.0016 (4)
C4	0.0434 (7)	0.0249 (6)	0.0357 (7)	−0.0014 (5)	0.0023 (5)	0.0003 (5)
C5	0.0335 (6)	0.0231 (5)	0.0343 (6)	0.0006 (4)	0.0004 (5)	−0.0012 (4)
C6	0.0384 (7)	0.0287 (6)	0.0443 (8)	0.0004 (5)	0.0028 (6)	−0.0077 (5)
C7	0.0375 (7)	0.0363 (6)	0.0460 (7)	−0.0018 (5)	0.0032 (6)	−0.0002 (5)
O1	0.0390 (5)	0.0296 (5)	0.0564 (6)	−0.0040 (4)	−0.0023 (4)	−0.0028 (4)
O2	0.0485 (6)	0.0379 (5)	0.0550 (6)	0.0090 (4)	0.0188 (5)	0.0089 (4)

Geometric parameters (Å, º)

C1—C3^{i}	1.3787 (17)	C5—C6	1.5183 (17)
C1—C2	1.3838 (17)	C5—H5	0.9800
C1—H1	0.9300	C6—O1	1.4203 (18)
C2—C3	1.3819 (17)	C6—H6A	0.9700
C2—H2	0.9300	C6—H6B	0.9700
C3—C4	1.5078 (18)	C7—O2	1.4190 (18)
C4—C5	1.5285 (18)	C7—H7A	0.9700
C4—H4A	0.9700	C7—H7B	0.9700
C4—H4B	0.9700	O1—H1A	0.820 (16)
C5—C7	1.513 (2)	O2—H2A	0.820 (17)
C3^{i}—C1—C2	121.33 (11)	C7—C5—H5	107.8
C3^{i}—C1—H1	119.3	C6—C5—H5	107.8
C2—C1—H1	119.3	C4—C5—H5	107.8
C3—C2—C1	121.04 (12)	O1—C6—C5	112.98 (10)
C3—C2—H2	119.5	O1—C6—H6A	109.0
C1—C2—H2	119.5	C5—C6—H6A	109.0
C1^{i}—C3—C2	117.63 (11)	O1—C6—H6B	109.0
C1^{i}—C3—C4	121.85 (11)	C5—C6—H6B	109.0
C2—C3—C4	120.51 (11)	H6A—C6—H6B	107.8
C3—C4—C5	113.62 (10)	O2—C7—C5	113.21 (11)
C3—C4—H4A	108.8	O2—C7—H7A	108.9
C5—C4—H4A	108.8	C5—C7—H7A	108.9
C3—C4—H4B	108.8	O2—C7—H7B	108.9
C5—C4—H4B	108.8	C5—C7—H7B	108.9
H4A—C4—H4B	107.7	H7A—C7—H7B	107.7
C7—C5—C6	110.72 (10)	C6—O1—H1A	109.5 (12)

C7—C5—C4	110.41 (10)	C7—O2—H2A	109.5 (12)
C6—C5—C4	112.03 (10)		
C3^{i}—C1—C2—C3	0.2 (2)	C3—C4—C5—C6	165.14 (10)
C1—C2—C3—C1^{i}	−0.2 (2)	C7—C5—C6—O1	173.08 (10)
C1—C2—C3—C4	178.72 (12)	C4—C5—C6—O1	−63.19 (14)
C1^{i}—C3—C4—C5	111.75 (15)	C6—C5—C7—O2	−65.22 (13)
C2—C3—C4—C5	−67.11 (15)	C4—C5—C7—O2	170.13 (9)
C3—C4—C5—C7	−70.96 (13)		

Symmetry code: (i) −*x*+2, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···O2^{ii}	0.820 (16)	1.906 (17)	2.7254 (17)	177.4 (17)
O2—H2*A*···O1^{iii}	0.820 (17)	1.943 (17)	2.7612 (17)	175.5 (17)

Symmetry codes: (ii) *x*+1/2, *y*, −*z*+3/2; (iii) −*x*+2, *y*−1/2, −*z*+3/2.