organic compounds

(1S*,2S*)-1,2-Di-*tert*-butylglycol

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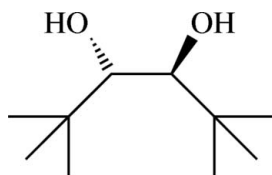Correspondence e-mail: kluef@cup.uni-muenchen.de

In the crystal structure of the title compound, $C_{10}H_{22}O_2$, co-operative chains of O—H···O hydrogen bonds are established by intra- as well as intermolecular interactions. These hydrogen bonds connect the molecules into infinite strands along [100], with a binary level graph-set descriptor $C_2^2(4)$. Excluding the H atoms on the hydroxy groups, the molecule shows non-crystallographic C_2 symmetry.

Related literature

The compound was synthesized according to a published procedure (Boehrer *et al.*, 1997). For the crystal structures of other ethane-1,2-diol derivatives with bulky substituents, see: Betz & Klüfers (2007); Allscher *et al.* (2008). For graph-set descriptors, see: Etter *et al.* (1990); Bernstein *et al.* (1995).

Experimental

Crystal data

$C_{10}H_{22}O_2$	c = 6.9771 (3) Å
M_r = 174.28	V = 1118.23 (8) Å^3
Orthorhombic, $P2_12_12$	Z = 4
a = 9.7799 (3) Å	Mo $K\alpha$ radiation
b = 16.3879 (7) Å	μ = 0.07 mm^{-1}

T = 200 (2) K	0.30 × 0.09 × 0.02 mm

Data collection

Nonius KappaCCD diffractometer	1490 independent reflections
Absorption correction: none	1253 reflections with $I > 2\sigma(I)$
8640 measured reflections	R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041	117 parameters
$wR(F^2)$ = 0.109	H-atom parameters constrained
S = 1.05	$\Delta\rho_{max}$ = 0.15 e Å^{-3}
1490 reflections	$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H811···O2^i	0.84	1.93	2.7721 (16)	176
O2—H821···O1	0.84	1.97	2.5129 (16)	121

Symmetry code: (i) $x - \frac{1}{2}, -y + \frac{1}{2}, -z + 2$.

Data collection: *COLLECT* (Nonius, 2004); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2332).

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supporting information

(1*S**,2*S**)-1,2-Di-*tert*-butylglycol

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S1. Comment

The title compound was synthesized as a potential chelating ligand with sterically demanding substituents on the carbon backbone to estimate the influence of steric pretense on coordination reactions with different central atoms.

In the molecule (Fig. 1) bond lengths and angles are found in the range apparent for other vicinal diols bearing sterically more demanding substituents (Betz & Klüfers, 2007, Allscher *et al.*, 2008). While the *tert*-butyl groups adopt a staggered conformation with respect to the hydroxy groups, the O atoms are present in a nearly eclipsed arrangement. The reason for this unfavourable conformation becomes evident when examining intermolecular contacts.

In the crystal structure, inter- and intramolecular hydrogen bonds are present which connect the molecules into strands along [1 0 0] (Fig. 2). The bulky hydrophobic *tert*-butyl groups encase this strand of hydroxyl groups. The hydrogen bonds form cooperative chains. In terms of graph-set analysis (Etter *et al.*, 1990; Bernstein *et al.*, 1995) the descriptor for these chains on the binary level is $C^2_2(4)$.

Excluding the H atoms on the hydroxy groups, the molecule shows non-crystallographic C_2 symmetry.

The molecular packing of the title compound is shown in Figure 3.

S2. Experimental

The title compound was prepared according to a published procedure (Boehrer *et al.*, 1997). Crystals suitable for X-ray studies were obtained upon recrystallization from boiling toluene.

S3. Refinement

Due to the absence of a strong anomalous scatterer, the absolute structure parameter, which is 0.982 with an estimated standard deviation of 1.241 for the unmerged data set, is meaningless. Thus 1056 Friedel opposites have been merged and the absolute configuration has been arbitrarily chosen.

Carbon-bound as well as oxygen-bound H atoms were placed in calculated positions (C—H 1.00 Å for CH-groups, C—H 0.98 Å for methyl groups and O—H 0.84 Å for hydroxy groups) and were included in the refinement in the riding model approximation, with $U(H)$ set to $1.2U_{eq}(C)$ for the CH-groups and $1.5U_{eq}(C)$ for methyl groups and $1.5U_{eq}(O)$ for the hydroxy groups.
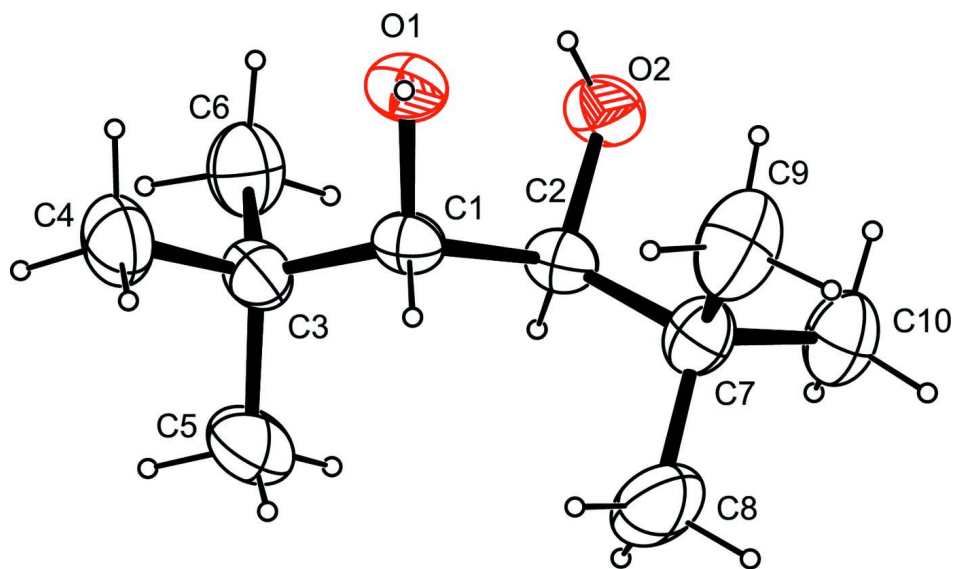
Figure 1

The molecular structure of the title compound, with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level) for non-H atoms.
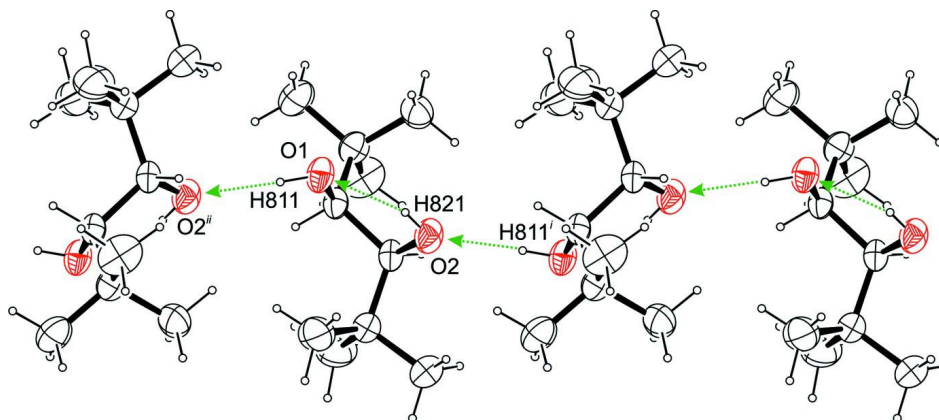
Figure 2

Hydrogen bonds in the crystal structure of the title compound, viewed along [0 0 1]. Underlying hydrogen bonds are not illustrated for clarity. Symmetry codes: $^{i}x + 1/2, -y + 1/2, -z + 2$; $^{ii}x - 1/2, -y + 1/2, -z + 2$.
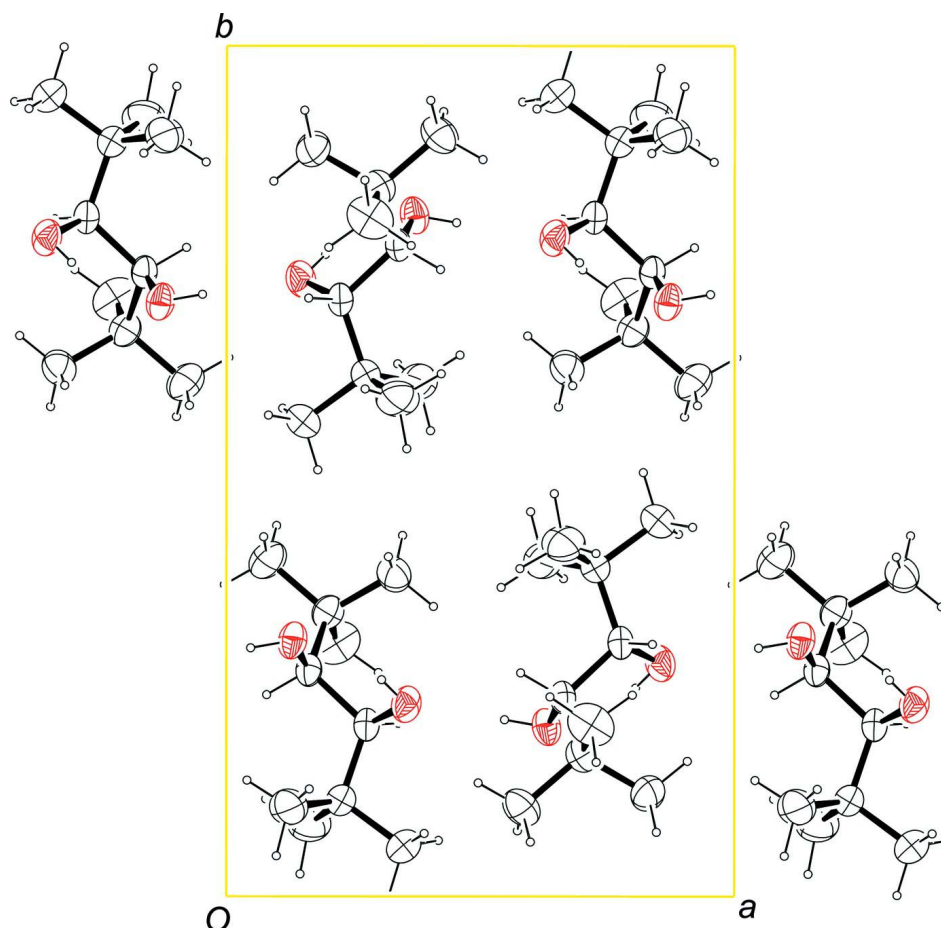
Figure 3

The packing of the title compound, viewed along [0 0 - 1].

(1S*,2S*)-1,2-Di-*tert*-butylglycol

Crystal data

$C_{10}H_{22}O_2$	$F(000) = 392$
$M_r = 174.28$	$D_x = 1.035$ Mg m^{-3}
Orthorhombic, $P2_12_12$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: P 2 2ab	Cell parameters from 13560 reflections
$a = 9.7799$ (3) Å	$\theta = 3.1$–27.5°
$b = 16.3879$ (7) Å	$\mu = 0.07$ mm^{-1}
$c = 6.9771$ (3) Å	$T = 200$ K
$V = 1118.23$ (8) Å^3	Rod, colourless
$Z = 4$	$0.30 \times 0.09 \times 0.02$ mm

Data collection

Nonius KappaCCD diffractometer	1490 independent reflections
Radiation source: rotating anode	1253 reflections with $I > 2\sigma(I)$
MONTEL, graded multilayered X-ray optics monochromator	$R_{int} = 0.040$
	$\theta_{max} = 27.5°$, $\theta_{min} = 3.2°$
CCD; rotation images; thick slices scans	$h = -12 \rightarrow 11$
8640 measured reflections	$k = -19 \rightarrow 21$
	$l = -9 \rightarrow 8$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.041$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.109$	
$S = 1.05$	H-atom parameters constrained
1490 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0621P)^2 + 0.0637P]$
117 parameters	where $P = (F_o^2 + 2F_c^2)/3$
0 restraints	$(\Delta/\sigma)_{max} < 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.15$ e Å^{-3}
	$\Delta\rho_{min} = -0.15$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\mathring{A}^2$)

	x	y	z	U_{iso}*/U_{eq}
O1	0.13030 (11)	0.30082 (8)	0.96003 (18)	0.0402 (3)
H811	0.0476	0.2922	0.9857	0.060*
O2	0.35411 (12)	0.22582 (8)	0.9762 (2)	0.0437 (4)
H821	0.3054	0.2548	1.0484	0.066*
C1	0.16231 (16)	0.26595 (10)	0.7782 (2)	0.0330 (4)
H1	0.0792	0.2371	0.7292	0.040*
C2	0.27611 (16)	0.20174 (10)	0.8121 (2)	0.0332 (4)
H2	0.3386	0.2035	0.6988	0.040*
C3	0.19990 (19)	0.33399 (11)	0.6353 (3)	0.0393 (4)
C4	0.0811 (2)	0.39538 (13)	0.6286 (3)	0.0551 (6)
H41	0.0731	0.4228	0.7530	0.083*
H42	−0.0042	0.3665	0.6001	0.083*
H43	0.0987	0.4360	0.5285	0.083*
C5	0.2176 (3)	0.29796 (15)	0.4358 (3)	0.0628 (6)
H51	0.2281	0.3422	0.3424	0.094*
H52	0.1370	0.2653	0.4031	0.094*
H53	0.2992	0.2632	0.4335	0.094*
C6	0.3298 (2)	0.37869 (13)	0.6961 (3)	0.0516 (5)
H61	0.4075	0.3409	0.6917	0.077*
H62	0.3188	0.3994	0.8270	0.077*
H63	0.3466	0.4244	0.6087	0.077*
C7	0.22584 (18)	0.11294 (12)	0.8338 (3)	0.0409 (5)
C8	0.1634 (3)	0.08512 (14)	0.6441 (4)	0.0687 (7)
H81	0.2274	0.0967	0.5394	0.103*
H82	0.0776	0.1145	0.6218	0.103*
H83	0.1453	0.0264	0.6494	0.103*
C9	0.1230 (2)	0.10514 (15)	0.9965 (4)	0.0656 (7)
H91	0.0986	0.0476	1.0138	0.098*
H92	0.0407	0.1366	0.9653	0.098*
H93	0.1634	0.1262	1.1150	0.098*
C10	0.3495 (2)	0.05872 (12)	0.8749 (4)	0.0542 (5)
H101	0.3896	0.0740	0.9985	0.081*
H102	0.4176	0.0658	0.7733	0.081*
H103	0.3204	0.0015	0.8793	0.081*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0321 (6)	0.0536 (8)	0.0349 (6)	0.0056 (6)	0.0041 (5)	−0.0067 (6)
O2	0.0344 (6)	0.0518 (8)	0.0448 (7)	0.0046 (5)	−0.0136 (6)	−0.0073 (6)
C1	0.0260 (7)	0.0416 (9)	0.0314 (8)	0.0013 (7)	−0.0020 (7)	−0.0047 (7)
C2	0.0251 (7)	0.0428 (9)	0.0317 (8)	0.0007 (7)	−0.0004 (6)	−0.0032 (7)
C3	0.0397 (9)	0.0446 (10)	0.0334 (9)	0.0051 (8)	−0.0020 (8)	0.0026 (8)
C4	0.0548 (11)	0.0537 (12)	0.0568 (12)	0.0140 (10)	−0.0059 (11)	0.0094 (11)
C5	0.0841 (15)	0.0716 (15)	0.0327 (10)	0.0129 (13)	0.0028 (10)	0.0025 (10)
C6	0.0452 (11)	0.0493 (11)	0.0602 (13)	−0.0051 (9)	0.0029 (10)	0.0121 (10)
C7	0.0341 (9)	0.0374 (9)	0.0513 (11)	0.0025 (7)	0.0012 (8)	−0.0015 (8)
C8	0.0733 (15)	0.0485 (12)	0.0843 (18)	−0.0004 (12)	−0.0263 (14)	−0.0208 (12)
C9	0.0580 (13)	0.0499 (12)	0.0889 (18)	−0.0016 (10)	0.0295 (12)	0.0095 (12)
C10	0.0463 (10)	0.0435 (11)	0.0729 (14)	0.0087 (9)	−0.0010 (11)	0.0023 (11)

Geometric parameters (Å, º)

O1—C1	1.426 (2)	C5—H53	0.980
O1—H811	0.840	C6—H61	0.980
O2—C2	1.431 (2)	C6—H62	0.980
O2—H821	0.840	C6—H63	0.980
C1—C3	1.540 (3)	C7—C9	1.522 (3)
C1—C2	1.550 (2)	C7—C8	1.527 (3)
C1—H1	1.000	C7—C10	1.528 (3)
C2—C7	1.543 (3)	C8—H81	0.980
C2—H2	1.000	C8—H82	0.980
C3—C5	1.522 (3)	C8—H83	0.980
C3—C6	1.527 (3)	C9—H91	0.980
C3—C4	1.538 (2)	C9—H92	0.980
C4—H41	0.980	C9—H93	0.980
C4—H42	0.980	C10—H101	0.980
C4—H43	0.980	C10—H102	0.980
C5—H51	0.980	C10—H103	0.980
C5—H52	0.980		
C1—O1—H811	109.5	H52—C5—H53	109.5
C2—O2—H821	109.5	C3—C6—H61	109.5
O1—C1—C3	109.77 (14)	C3—C6—H62	109.5
O1—C1—C2	107.08 (13)	H61—C6—H62	109.5
C3—C1—C2	114.77 (13)	C3—C6—H63	109.5
O1—C1—H1	108.3	H61—C6—H63	109.5
C3—C1—H1	108.3	H62—C6—H63	109.5
C2—C1—H1	108.3	C9—C7—C8	110.91 (18)
O2—C2—C7	110.58 (14)	C9—C7—C10	109.53 (17)
O2—C2—C1	108.52 (13)	C8—C7—C10	107.79 (17)
C7—C2—C1	115.24 (14)	C9—C7—C2	111.26 (16)
O2—C2—H2	107.4	C8—C7—C2	108.91 (16)

C7—C2—H2	107.4	C10—C7—C2	108.35 (14)
C1—C2—H2	107.4	C7—C8—H81	109.5
C5—C3—C6	110.22 (19)	C7—C8—H82	109.5
C5—C3—C4	108.20 (17)	H81—C8—H82	109.5
C6—C3—C4	108.86 (16)	C7—C8—H83	109.5
C5—C3—C1	109.78 (16)	H81—C8—H83	109.5
C6—C3—C1	111.47 (15)	H82—C8—H83	109.5
C4—C3—C1	108.22 (14)	C7—C9—H91	109.5
C3—C4—H41	109.5	C7—C9—H92	109.5
C3—C4—H42	109.5	H91—C9—H92	109.5
H41—C4—H42	109.5	C7—C9—H93	109.5
C3—C4—H43	109.5	H91—C9—H93	109.5
H41—C4—H43	109.5	H92—C9—H93	109.5
H42—C4—H43	109.5	C7—C10—H101	109.5
C3—C5—H51	109.5	C7—C10—H102	109.5
C3—C5—H52	109.5	H101—C10—H102	109.5
H51—C5—H52	109.5	C7—C10—H103	109.5
C3—C5—H53	109.5	H101—C10—H103	109.5
H51—C5—H53	109.5	H102—C10—H103	109.5
O1—C1—C2—O2	−28.26 (17)	O1—C1—C3—C4	−55.45 (18)
C3—C1—C2—O2	93.86 (17)	C2—C1—C3—C4	−176.09 (15)
O1—C1—C2—C7	96.34 (17)	O2—C2—C7—C9	66.46 (19)
C3—C1—C2—C7	−141.55 (15)	C1—C2—C7—C9	−57.1 (2)
O1—C1—C3—C5	−173.34 (16)	O2—C2—C7—C8	−170.99 (15)
C2—C1—C3—C5	66.0 (2)	C1—C2—C7—C8	65.5 (2)
O1—C1—C3—C6	64.23 (18)	O2—C2—C7—C10	−54.00 (19)
C2—C1—C3—C6	−56.4 (2)	C1—C2—C7—C10	−177.51 (16)

Hydrogen-bond geometry (Å, º)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O1—H811⋯O2[i]	0.84	1.93	2.7721 (16)	176
O2—H821⋯O1	0.84	1.97	2.5129 (16)	121

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