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(2,3-Dihydro-1,4-benzodioxin-2-yl)-methanol

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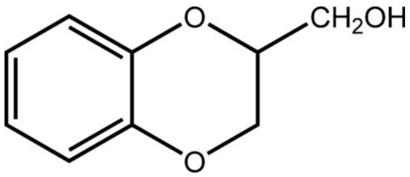
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.041; wR factor = 0.108; data-to-parameter ratio = 16.8.

In the title compound, $C_9H_{10}O_3$, the heterocyclic ring has a half-chair conformation. The –CH_2OH substituent is attached equatorially with respect to the heterocyclic ring. In the crystal structure, the molecules are linked by O—H⋯O hydrogen bonds, with the hydroxyl O atom functioning both as donor and acceptor. This results in the formation of C(2) helical chains running along the fourfold screw axes.

Related literature

In closely related salt structures, there are chains running along a twofold screw axis built up by hydrogen-bond interactions, involving the ammonium groups NH_3^+ and the carboxylate O atoms (Marchini *et al.*, 2005).

For related literature, see: Bernstein *et al.* (1995); Bolchi *et al.* (2003); Cremer & Pople (1975); Guillaumet (1996).



Experimental

Crystal data

$C_9H_{10}O_3$	Z = 16
M_r = 166.17	Mo $K\alpha$ radiation
Tetragonal, $I4_1/a$	μ = 0.10 mm^{-1}
a = 22.5161 (10) Å	T = 100 (2) K
c = 6.3400 (6) Å	0.55 × 0.23 × 0.15 mm
V = 3214.2 (4) Å^3	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	9739 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)	1883 independent reflections
T_{min} = 0.955, T_{max} = 0.985	1716 reflections with $I > 2\sigma(I)$
	R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.108	$\Delta\rho_{max}$ = 0.35 e Å^{-3}
S = 1.03	$\Delta\rho_{min}$ = −0.21 e Å^{-3}
1883 reflections	
112 parameters	
1 restraint	

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O3—H3⋯O3^i	0.840 (14)	1.801 (14)	2.6348 (11)	172.1 (17)

Symmetry code: (i) $-y+\frac{5}{4}, x-\frac{1}{4}, z-\frac{1}{4}$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: publCIF (Westrip, 2007).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2014).

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supplementary materials

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(2,3-Dihydro-1,4-benzodioxin-2-yl)methanol

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Comment

The 2,3-dihydro-1,4-benzodioxin ring system is present in a large number of structures of therapeutic agents possessing important biological activities (Guillaumet, 1996). As part of our ongoing investigation of this kind of system, we recently prepared the title compound, (I), and present here its crystal structure.

The structure of (I) is shown in Fig. 1. The six-membered ring containing O atoms adopts a half-chair conformation. The Cremer & Pople (1975) puckering parameters for this ring are Q = 0.5095 (11), θ = 51.72 (13)° and φ = 151.75 (14)°. The methanol group attached to C7 is in an equatorial position.

In the crystal structure, molecules form helical chains along *c*, involving their hydroxyl groups both as donors and acceptors of hydrogen bonds (Table 2), with graph set *C*(2) (Bernstein *et al.*, 1995), *via* crystallographic fourfold screw axes (Fig. 2).

Experimental

The title compound was obtained as follows. Catechol (1.0 equivalent) was added at room temperature to a solution of K_2CO_3 (2.5 equivalents) in distilled water and the resulting solution was stirred for 30 min. Epichlorohydrin (1.3 equivalents) was then added dropwise and warmed at 343 K for 6 h. After cooling to room temperature, the reaction mixture was diluted and extracted (CH_2Cl_2), the organic layer dried (Na_2SO_4), filtered and concentrated, and the crude product purified by flash chromatography (gradient elution EtOAc–hexane 1:5 → 1:1.5). The title compound was obtained (46% yield) as a white solid [m.p. 360–361 K (literature value 362.35 K; Bolchi *et al.*, 2003)]. Suitable crystals of (I) were obtained after dissolving the compound in the minimum volume of CH_2Cl_2. A vial with a screw top allowed the slow evaporation of the solvent at room temperature to produce colourless crystals.

Refinement

The hydroxy H atom was located in a difference map and refined as riding, with O—H = 0.84 Å and with U_{iso}(H) = 1.2U_{eq}(O). Other H atoms were positioned geometrically and treated as riding, with C—H = 0.95–0.99 Å and with U_{iso}(H) = 1.2U_{eq}(C).

Figures

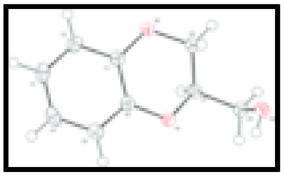

Fig. 1. The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented as spheres of arbitrary radii.

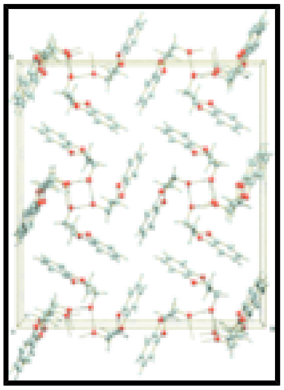

Fig. 2. A view, down the *c* axis, of the molecular packing of (I). The intermolecular hydrogen bonds are shown as dashed lines.

(2,3-Dihydro-1,4-benzodioxin-2-yl)methanol

Crystal data

$C_9H_{10}O_3$

M_r = 166.17

Tetragonal, $I4_1/a$

Hall symbol: -I 4ad

a = 22.5161 (10) Å

b = 22.5161 (10) Å

c = 6.3400 (6) Å

α = 90°

β = 90°

γ = 90°

V = 3214.2 (4) Å^3

Z = 16

F_{000} = 1408

D_x = 1.374 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71073 Å

Cell parameters from 4441 reflections

θ = 1.8–28.3°

μ = 0.10 mm^{-1}

T = 100 (2) K

Prism, colourless

0.55 × 0.23 × 0.15 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer

Monochromator: graphite

T = 100(2) K

φ and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 2004)

$T_{\min}$ = 0.955, $T_{\max}$ = 0.985

9739 measured reflections

1883 independent reflections

1716 reflections with $I > 2\sigma(I)$

R_{int} = 0.024

$\theta_{\max}$ = 28.3°

$\theta_{\min}$ = 1.8°

$h = -25 \rightarrow 29$

$k = -18 \rightarrow 29$

$l = -8 \rightarrow 7$

Refinement

Refinement on F^2

Least-squares matrix: full

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$

$wR(F^2) = 0.108$

$S = 1.03$

1883 reflections

112 parameters

1 restraint

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

$w = 1/[\sigma^2(F_o^2) + (0.0545P)^2 + 3.1875P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.35$ e Å^{-3}

$\Delta\rho_{min} = -0.21$ e Å^{-3}

Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.79405 (5)	0.30835 (5)	0.4847 (2)	0.0185 (3)
C2	0.82759 (6)	0.27676 (6)	0.6307 (2)	0.0235 (3)
H2	0.8132	0.2715	0.7703	0.028*
C3	0.88200 (6)	0.25297 (6)	0.5731 (2)	0.0258 (3)
H3A	0.9047	0.2312	0.6728	0.031*
C4	0.90330 (6)	0.26102 (6)	0.3693 (2)	0.0258 (3)
H4	0.9410	0.2454	0.3308	0.031*
C5	0.86967 (6)	0.29177 (6)	0.2219 (2)	0.0226 (3)
H5	0.8844	0.2971	0.0827	0.027*
C6	0.81441 (5)	0.31479 (5)	0.2776 (2)	0.0177 (3)
O1	0.78152 (4)	0.34302 (4)	0.12524 (14)	0.0197 (2)
C7	0.71960 (5)	0.34921 (5)	0.1827 (2)	0.0194 (3)
H7	0.7008	0.3090	0.1812	0.023*
C8	0.71610 (6)	0.37380 (6)	0.4038 (2)	0.0233 (3)
H8A	0.7386	0.4115	0.4125	0.028*
H8B	0.6742	0.3821	0.4408	0.028*
O2	0.74068 (4)	0.33105 (4)	0.54951 (14)	0.0236 (2)
C9	0.68964 (6)	0.38742 (6)	0.0181 (2)	0.0214 (3)
H9A	0.6953	0.3689	−0.1221	0.026*
H9B	0.6465	0.3885	0.0473	0.026*
O3	0.71146 (4)	0.44630 (4)	0.01122 (16)	0.0256 (2)
H3	0.7420 (7)	0.4480 (8)	−0.065 (3)	0.031*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0192 (6)	0.0155 (5)	0.0208 (6)	−0.0012 (4)	−0.0018 (5)	−0.0021 (4)
C2	0.0280 (7)	0.0212 (6)	0.0213 (6)	−0.0025 (5)	−0.0053 (5)	−0.0003 (5)
C3	0.0253 (6)	0.0196 (6)	0.0325 (7)	0.0001 (5)	−0.0132 (5)	−0.0012 (5)
C4	0.0162 (6)	0.0227 (6)	0.0385 (8)	0.0011 (5)	−0.0045 (5)	−0.0059 (5)
C5	0.0186 (6)	0.0224 (6)	0.0268 (7)	−0.0018 (5)	0.0008 (5)	−0.0026 (5)
C6	0.0178 (6)	0.0145 (5)	0.0209 (6)	−0.0021 (4)	−0.0019 (4)	−0.0011 (4)
O1	0.0177 (4)	0.0227 (4)	0.0186 (4)	0.0020 (3)	0.0017 (3)	0.0023 (3)
C7	0.0169 (6)	0.0201 (6)	0.0211 (6)	0.0007 (4)	0.0018 (5)	0.0000 (5)
C8	0.0245 (6)	0.0255 (6)	0.0198 (6)	0.0068 (5)	0.0011 (5)	0.0004 (5)
O2	0.0244 (5)	0.0286 (5)	0.0177 (5)	0.0070 (4)	0.0026 (4)	0.0025 (4)
C9	0.0215 (6)	0.0229 (6)	0.0198 (6)	0.0018 (5)	−0.0003 (5)	0.0008 (5)
O3	0.0272 (5)	0.0195 (5)	0.0299 (5)	0.0040 (4)	0.0082 (4)	0.0016 (4)

Geometric parameters (Å, °)

C1—O2	1.3689 (15)	O1—C7	1.4477 (14)
C1—C2	1.3906 (17)	C7—C8	1.5091 (18)
C1—C6	1.3981 (18)	C7—C9	1.5118 (17)
C2—C3	1.3861 (19)	C7—H7	1.0000
C2—H2	0.9500	C8—O2	1.4443 (15)
C3—C4	1.390 (2)	C8—H8A	0.9900
C3—H3A	0.9500	C8—H8B	0.9900
C4—C5	1.3878 (19)	C9—O3	1.4146 (15)
C4—H4	0.9500	C9—H9A	0.9900
C5—C6	1.3934 (17)	C9—H9B	0.9900
C5—H5	0.9500	O3—H3	0.840 (14)
C6—O1	1.3732 (15)		

O2—C1—C2	117.89 (11)	O1—C7—C9	108.11 (10)
O2—C1—C6	122.08 (11)	C8—C7—C9	114.17 (10)
C2—C1—C6	120.01 (11)	O1—C7—H7	108.6
C3—C2—C1	120.15 (13)	C8—C7—H7	108.6
C3—C2—H2	119.9	C9—C7—H7	108.6
C1—C2—H2	119.9	O2—C8—C7	109.24 (10)
C2—C3—C4	119.97 (12)	O2—C8—H8A	109.8
C2—C3—H3A	120.0	C7—C8—H8A	109.8
C4—C3—H3A	120.0	O2—C8—H8B	109.8
C5—C4—C3	120.18 (12)	C7—C8—H8B	109.8
C5—C4—H4	119.9	H8A—C8—H8B	108.3
C3—C4—H4	119.9	C1—O2—C8	113.15 (10)
C4—C5—C6	120.14 (13)	O3—C9—C7	113.55 (11)
C4—C5—H5	119.9	O3—C9—H9A	108.8
C6—C5—H5	119.9	C7—C9—H9A	108.9
O1—C6—C5	118.39 (11)	O3—C9—H9B	108.9
O1—C6—C1	122.12 (11)	C7—C9—H9B	108.9

C5—C6—C1	119.49 (11)	H9A—C9—H9B	107.7
C6—O1—C7	112.76 (9)	C9—O3—H3	110.1 (12)
O1—C7—C8	108.63 (10)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3···O3[i]	0.840 (14)	1.801 (14)	2.6348 (11)	172.1 (17)

Symmetry codes: (i) −*y*+5/4, *x*−1/4, *z*−1/4.

Fig. 1

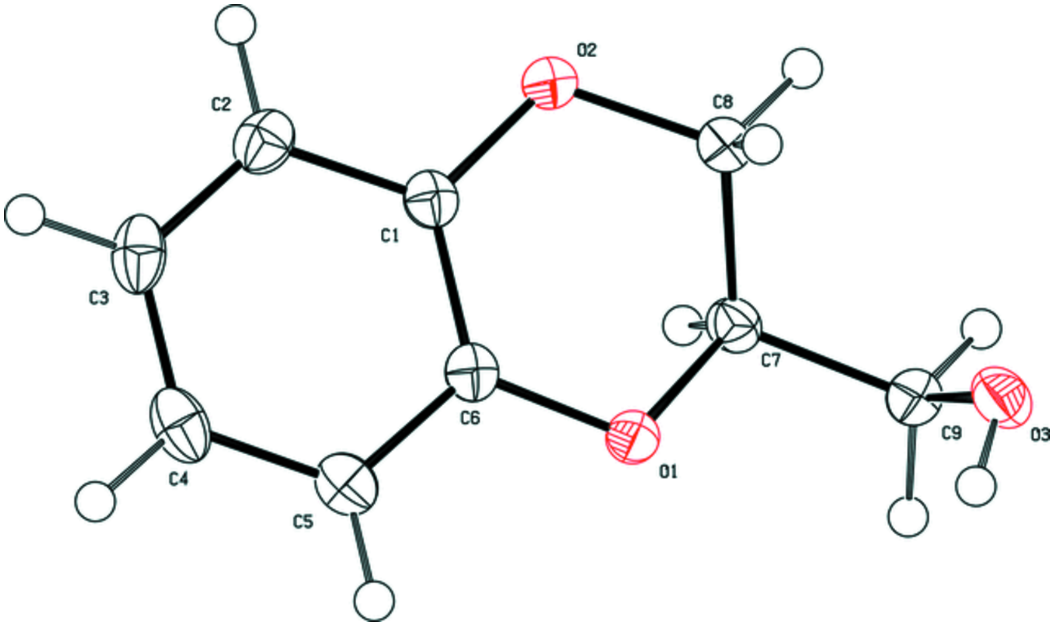

Fig. 2