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2,6-Dimethyl-9-oxabicyclo[3.3.1]nonane-*endo*-2,*endo*-6-diol

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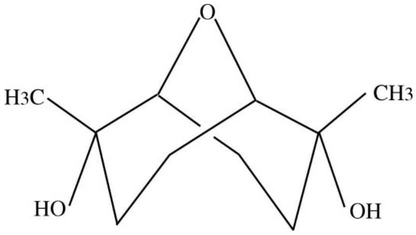
Key indicators: single-crystal X-ray study; T = 170 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.122; data-to-parameter ratio = 18.3.

The structure of the title compound, $C_{10}H_{18}O_3$, can be described in the space group *Cc* with two independent molecules in the asymmetric unit almost related by a twofold axis due to the different orientations of the H atoms of the hydroxyl groups. However, in the space group *C*2/*c* this pseudo-twofold axis is a crystallographic one and these H atoms appear to be disordered. The same situation (Cambridge Structural Database, CSD) has been observed in the structure of the carbobicyclic derivative (CSD refcode SEWXAT), described in the space group *Pc* with two independent molecules, while in the thiobicyclic analogue (SEWXEX), the H atoms are disordered in the space group $P2_1/c$. In these three compounds, the hydroxyl groups are in equatorial positions and *anti* with respect to the ether atom bridge. The supramolecular structure of the title compound consists of layers where the hydroxyl groups are involved as donor and acceptor of hydrogen bonds [graph-set motif $R_4^4(8)$]. This layered structure shows a close resemblance to those of the carbo- and thiobicyclic analogues but is dissimilar to that of the *exo* diastereoisomer derivative, which has OH groups in a *syn* disposition with respect to the ether bridge (WASWAO).

Related literature

For the related structures [Cambridge Structural Database (CSD), Version 5.28; Allen, 2002] of the *endo*-2,*endo*-6-dihydroxy-2,6-dimethylbicyclo[3.3.1]nonane (CSD refcode SEWXAT) and *endo*-2,*endo*-6-dihydroxy-2,6-thiobicyclo-[3.3.1]nonane (CSD refcode SEWXEX) analogues, see: Hawkins *et al.* (1990). For the *exo* diastereoisomer (CSD refcode WASWAO) of the title compound, see: Pich *et al.* (1993). For *syn*-diol derivatives with inclusion behaviour, see: Kim *et al.* (2002).

For related literature, see: Bernstein *et al.* (1995); Carrasco (2001).



Experimental

Crystal data

$C_{10}H_{18}O_3$
M_r = 186.24
Monoclinic, $C2/c$
a = 10.8900 (2) Å
b = 9.9227 (2) Å
c = 18.8981 (3) Å
β = 104.658 (11)°
V = 1975.63 (12) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 170 (2) K
0.40 × 0.23 × 0.17 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: none
11200 measured reflections
2220 independent reflections
2113 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.122
S = 1.08
2220 reflections
121 parameters
H-atom parameters constrained
$\Delta\rho_{\rm max}$ = 0.37 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.27 e Å^{-3}

Table 1
Selected torsion angles (°).

O1—C1—C2—O2	−175.6 (1)	C1—C2—O2—H2	79
O1—C1—C2—C9	67.3 (1)	C1—C2—O2—H12	−39
O1—C5—C6—O3	−173.1 (1)	C5—C6—O3—H3	−56
O1—C5—C6—C10	68.4 (1)	C5—C6—O3—H13	−162

Table 2
Hydrogen-bond geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O2—H2⋯O2$^{\rm i}$	0.84	1.92	2.728 (2)	160
O2—H12⋯O3$^{\rm ii}$	0.84	1.96	2.687 (1)	145
O3—H3⋯O2$^{\rm iii}$	0.84	2.04	2.687 (1)	133
O3—H13⋯O3$^{\rm iv}$	0.84	2.02	2.738 (2)	143

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

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *WinGX* (Farrugia, 1999), *PLATON* (Spek, 2003) and *Mercury* (Macrae *et al.*, 2006).

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J. D. Martin (Instituto Investigaciones Qímicas, CSIC, Sevilla, Spain) for suggesting this study and providing the sample.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: YM2059).

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supplementary materials

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2,6-Dimethyl-9-oxabicyclo[3.3.1]nonane-*endo*-2,*endo*-6-diol

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Comment

In a project related with the synthesis of a universal template for resolution of racemic mixtures of secondary alcohols, the intermediate title compound was synthesized (Carrasco, 2001). On the other hand, the analogous *syn*-diols derivatives studied so far have proved to have inclusion behaviour (Kim *et al.*, 2002).

The two independent molecules of (I) in the *Cc* space group differ from each other only in the orientation of the H atoms of the hydroxyl groups. The molecules are almost related by a twofold axis which is a crystallographic one in the *C*2/*c* space group and, therefore, the H atoms are split into two positions (Figure 1). The same situation has been observed (Cambridge Crystallographic Database, Allen, 2002) in the structure of the *endo*-2,*endo*-6-dihydroxy-2,6-dimethylbicyclo(3.3.1)nonane compound described in the *Pc* group (SEWXAT refcode: Hawkins *et al.*, 1990) with a CH_2 group instead of the ether bridge while in the thio-bicycle derivative (SEWXEX refcode, $P2_1/c$ space group: Hawkins *et al.*, 1990) the H of the hydroxyl groups appear to be disordered.

The six-membered rings are in a slightly distorted chair conformation with the hydroxyl groups in equatorial position, *anti* with respect to the ether atom O1 (Table 1). These OH groups are involved in the formation of the $R_4^4(8)$ hydrogen bonding motif (Bernstein *et al.*, 1995) acting as both donor and acceptor of hydrogen bonds that results into sheets (Table 2, Figure 2 and 3). Weak C—H···O_{ether} contacts link the layers into a three-dimensional network (2.65, 3.613 (1)Å and 162° for the H···A, D···A distances and DH···A angle). This layered structure is similar to that of carbo-bicycle and thio-biclycle analogues (SEWXAT and SEWXEX) but dissimilar to that of the *exo* diastereoisomer derivative (OH groups in *syn* disposition with respect to the ether bridge (WASWAO, Pich *et al.*, 1993). These results are in agreement with the rules proposed by Kim *et al.*, (2002) to be fulfilled by the molecular structure of these derivatives to form supramolecular tubulant hydrogen-bonding networks.

Experimental

Compound (I) was synthesized (Carrasco, 2001) within a project related with the synthesis of a universal template for resolution of racemic mixtures of secondary alcohols and was crystallized from a mixture of acetone/n-hexane at 50%. No structural phase transition was detected when cooling the sample from room temperature to 170 K.

Refinement

Refinements were performed in the *Cc* and *C*2/*c* space groups. In the *C*2/*c* group, the H atom of the hydroxyl groups were split into two positions while in the *Cc* space group refinements were carried out with two independent molecules without disorder. The lack of suitable anomalous scatters did not allow us to reliably determine the absolute structure according to the Flack parameters = 0.3 (14) and, therefore, the Friedel pairs were merged. All hydrogen atoms were located on difference

Fourier maps and they were included in the refinement in the riding-model approximation with C—H = 0.98 (CH_3), 0.99 (CH_2), 1.00 (CH) and O—H = 0.84 Å with $U_{iso}(H) = 1.2U_{eq}(C,O)$.

Figures

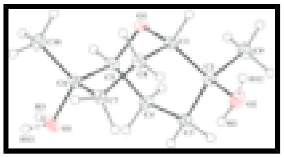

Fig. 1. Asymmetric unit showing displacement ellipsoids at the 30% probability level. Dashed lines represented the disordered H atoms.

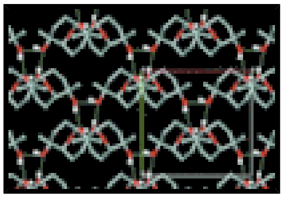

Fig. 2. Two-dimensional network showing the $R_4^4(8)$ and $R_4^4(36)$ rings. Hydrogen atoms not involved in the O—H···O hydrogen bonds have been omitted as well as the disorder.

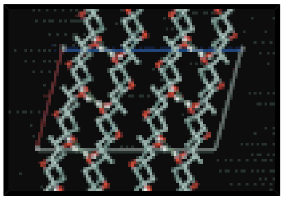

Fig. 3. Packing diagram showing two sheets perpendicular to the *ac* plane. Hydrogen atoms not involved in the O—H···O hydrogen bonds have omitted.

2,6-Dimethyl-9-oxabicyclo[3.3.1]nonane-*endo*-2,*endo*-6-diol

Crystal data

$C_{10}H_{18}O_3$	$F_{000} = 816$
$M_r = 186.24$	$D_x = 1.252$ Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71069 Å
Hall symbol: -C 2yc	Cell parameters from 11200 reflections
a = 10.8900 (2) Å	θ = 2.2–27.5°
b = 9.9227 (2) Å	μ = 0.09 mm^{-1}
c = 18.8981 (3) Å	*T* = 170 (2) K
β = 104.658 (11)°	Plate, colourless
V = 1975.63 (12) Å^3	0.40 × 0.23 × 0.17 mm
Z = 8	

Data collection

Nonius KappaCCD area-detector diffractometer	2220 independent reflections
Radiation source: fine-focus sealed tube	2113 reflections with $I > 2\sigma(I)$
Monochromator: horizontally mounted graphite crystal	$R_{int} = 0.032$
Detector resolution: 9 pixels mm^{-1}	$\theta_{max} = 27.5°$
T = 170(2) K	$\theta_{min} = 2.2°$

φ and ω scans
Absorption correction: none
11200 measured reflections
$h = -14 \rightarrow 14$
$k = -12 \rightarrow 12$
$l = -23 \rightarrow 24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.122$
$S = 1.08$
2220 reflections
121 parameters
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0645P)^2 + 1.3903P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.37$ e Å^{-3}
$\Delta\rho_{\min} = -0.27$ e Å^{-3}
Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.063 (6)

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}	Occ. (<1)
O1	0.14392 (7)	0.13828 (8)	0.05218 (4)	0.0205 (2)	
O2	−0.05943 (9)	0.06522 (9)	0.17721 (5)	0.0325 (3)	
H2	−0.0068	0.0669	0.2183	0.039*	0.50
H12	−0.0688	−0.0156	0.1637	0.039*	0.50
O3	0.39487 (9)	0.30199 (9)	0.19209 (5)	0.0329 (3)	
H3	0.4123	0.3624	0.1650	0.039*	0.50
H13	0.4693	0.2797	0.2137	0.039*	0.50
C1	0.08890 (10)	0.05681 (11)	0.09945 (6)	0.0197 (3)	
H1	0.0414	−0.0175	0.0685	0.024*	
C2	−0.01064 (10)	0.14124 (11)	0.12572 (6)	0.0199 (3)	
C3	0.04768 (10)	0.27108 (11)	0.16325 (6)	0.0203 (3)	

H3A	0.1006	0.2493	0.2127	0.024*
H3B	−0.0211	0.3321	0.1689	0.024*
C4	0.12935 (11)	0.34342 (11)	0.12003 (6)	0.0209 (3)
H4A	0.0730	0.3907	0.0780	0.025*
H4B	0.1818	0.4123	0.1518	0.025*
C5	0.21646 (10)	0.24856 (11)	0.09164 (6)	0.0183 (3)
H5	0.2496	0.3010	0.0552	0.022*
C6	0.33340 (10)	0.19199 (11)	0.14790 (6)	0.0194 (3)
C7	0.29534 (11)	0.08904 (12)	0.19901 (6)	0.0214 (3)
H7A	0.3715	0.0380	0.2250	0.026*
H7B	0.2623	0.1375	0.2361	0.026*
C8	0.19427 (11)	−0.01001 (11)	0.15796 (6)	0.0226 (3)
H8A	0.2358	−0.0796	0.1345	0.027*
H8B	0.1559	−0.0559	0.1937	0.027*
C9	−0.12245 (11)	0.17108 (14)	0.06051 (7)	0.0296 (3)
H9A	−0.1577	0.0862	0.0376	0.044*
H9B	−0.0934	0.2260	0.0248	0.044*
H9C	−0.1880	0.2202	0.0772	0.044*
C10	0.42431 (11)	0.13079 (13)	0.10697 (7)	0.0278 (3)
H10A	0.4471	0.1992	0.0751	0.042*
H10B	0.3830	0.0546	0.0773	0.042*
H10C	0.5012	0.0993	0.1423	0.042*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0249 (4)	0.0206 (4)	0.0156 (4)	−0.0001 (3)	0.0046 (3)	−0.0026 (3)
O2	0.0337 (5)	0.0271 (5)	0.0423 (5)	0.0021 (4)	0.0200 (4)	0.0106 (4)
O3	0.0296 (5)	0.0241 (5)	0.0412 (5)	−0.0056 (3)	0.0023 (4)	−0.0107 (4)
C1	0.0224 (5)	0.0162 (5)	0.0197 (5)	−0.0001 (4)	0.0037 (4)	−0.0020 (4)
C2	0.0212 (5)	0.0185 (5)	0.0201 (5)	0.0011 (4)	0.0052 (4)	0.0016 (4)
C3	0.0245 (5)	0.0170 (5)	0.0208 (5)	0.0025 (4)	0.0082 (4)	−0.0009 (4)
C4	0.0248 (6)	0.0147 (5)	0.0241 (5)	0.0034 (4)	0.0079 (4)	0.0014 (4)
C5	0.0221 (5)	0.0157 (5)	0.0174 (5)	0.0012 (4)	0.0057 (4)	0.0012 (4)
C6	0.0212 (5)	0.0156 (5)	0.0202 (5)	0.0002 (4)	0.0031 (4)	−0.0009 (4)
C7	0.0233 (5)	0.0200 (5)	0.0193 (5)	0.0026 (4)	0.0024 (4)	0.0036 (4)
C8	0.0240 (6)	0.0157 (5)	0.0273 (6)	0.0025 (4)	0.0051 (4)	0.0028 (4)
C9	0.0232 (6)	0.0329 (7)	0.0292 (6)	0.0049 (5)	0.0000 (5)	−0.0002 (5)
C10	0.0246 (6)	0.0287 (6)	0.0316 (6)	0.0063 (5)	0.0100 (5)	0.0023 (5)

Geometric parameters (Å, °)

O1—C5	1.4420 (13)	C4—H4A	0.9900
O1—C1	1.4427 (13)	C4—H4B	0.9900
O2—C2	1.4352 (13)	C5—C6	1.5433 (14)
O2—H2	0.8400	C5—H5	1.0000
O2—H12	0.8400	C6—C10	1.5280 (16)
O3—C6	1.4331 (13)	C6—C7	1.5332 (15)
O3—H3	0.8400	C7—C8	1.5320 (16)

O3—H13	0.8400	C7—H7A	0.9900
C1—C8	1.5287 (15)	C7—H7B	0.9900
C1—C2	1.5478 (15)	C8—H8A	0.9900
C1—H1	1.0000	C8—H8B	0.9900
C2—C9	1.5263 (16)	C9—H9A	0.9800
C2—C3	1.5292 (15)	C9—H9B	0.9800
C3—C4	1.5300 (15)	C9—H9C	0.9800
C3—H3A	0.9900	C10—H10A	0.9800
C3—H3B	0.9900	C10—H10B	0.9800
C4—C5	1.5269 (14)	C10—H10C	0.9800

C5—O1—C1	111.34 (8)	C4—C5—H5	106.6
C2—O2—H2	109.5	C6—C5—H5	106.6
C2—O2—H12	109.5	O3—C6—C10	109.31 (9)
C6—O3—H3	109.5	O3—C6—C7	107.54 (9)
C6—O3—H13	109.5	C10—C6—C7	111.54 (9)
O1—C1—C8	109.73 (9)	O3—C6—C5	107.86 (9)
O1—C1—C2	109.25 (8)	C10—C6—C5	108.85 (9)
C8—C1—C2	117.46 (9)	C7—C6—C5	111.64 (9)
O1—C1—H1	106.6	C8—C7—C6	112.49 (9)
C8—C1—H1	106.6	C8—C7—H7A	109.1
C2—C1—H1	106.6	C6—C7—H7A	109.1
O2—C2—C9	107.03 (9)	C8—C7—H7B	109.1
O2—C2—C3	108.05 (9)	C6—C7—H7B	109.1
C9—C2—C3	111.34 (9)	H7A—C7—H7B	107.8
O2—C2—C1	110.03 (9)	C1—C8—C7	113.55 (9)
C9—C2—C1	109.05 (9)	C1—C8—H8A	108.9
C3—C2—C1	111.25 (9)	C7—C8—H8A	108.9
C2—C3—C4	112.14 (9)	C1—C8—H8B	108.9
C2—C3—H3A	109.2	C7—C8—H8B	108.9
C4—C3—H3A	109.2	H8A—C8—H8B	107.7
C2—C3—H3B	109.2	C2—C9—H9A	109.5
C4—C3—H3B	109.2	C2—C9—H9B	109.5
H3A—C3—H3B	107.9	H9A—C9—H9B	109.5
C5—C4—C3	113.28 (9)	C2—C9—H9C	109.5
C5—C4—H4A	108.9	H9A—C9—H9C	109.5
C3—C4—H4A	108.9	H9B—C9—H9C	109.5
C5—C4—H4B	108.9	C6—C10—H10A	109.5
C3—C4—H4B	108.9	C6—C10—H10B	109.5
H4A—C4—H4B	107.7	H10A—C10—H10B	109.5
O1—C5—C4	109.98 (8)	C6—C10—H10C	109.5
O1—C5—C6	109.20 (8)	H10A—C10—H10C	109.5
C4—C5—C6	117.37 (9)	H10B—C10—H10C	109.5
O1—C5—H5	106.6		

C5—O1—C1—C8	−64.28 (10)	O1—C5—C6—O3	−173.1 (1)
C5—O1—C1—C2	65.83 (11)	C4—C5—C6—O3	−47.12 (12)
O1—C1—C2—O2	−175.6 (1)	O1—C5—C6—C10	68.4 (1)
C8—C1—C2—O2	−49.86 (13)	C4—C5—C6—C10	−165.62 (9)
O1—C1—C2—C9	67.3 (1)	O1—C5—C6—C7	−55.19 (11)

C8—C1—C2—C9	−166.96 (10)	C4—C5—C6—C7	70.81 (12)
O1—C1—C2—C3	−55.91 (11)	O3—C6—C7—C8	162.07 (9)
C8—C1—C2—C3	69.86 (12)	C10—C6—C7—C8	−78.10 (12)
O2—C2—C3—C4	166.59 (9)	C5—C6—C7—C8	43.94 (12)
C9—C2—C3—C4	−76.15 (12)	O1—C1—C8—C7	51.38 (12)
C1—C2—C3—C4	45.70 (12)	C2—C1—C8—C7	−74.14 (12)
C2—C3—C4—C5	−44.26 (12)	C6—C7—C8—C1	−42.51 (13)
C1—O1—C5—C4	−63.63 (10)	C1—C2—O2—H2	79
C1—O1—C5—C6	66.52 (10)	C1—C2—O2—H12	−39
C3—C4—C5—O1	52.01 (11)	C5—C6—O3—H3	−56
C3—C4—C5—C6	−73.60 (12)	C5—C6—O3—H13	−162

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O2$^{\mathrm{i}}$	0.84	1.92	2.728 (2)	160
O2—H12···O3$^{\mathrm{ii}}$	0.84	1.96	2.687 (1)	145
O3—H3···O2$^{\mathrm{iii}}$	0.84	2.04	2.687 (1)	133
O3—H13···O3$^{\mathrm{iv}}$	0.84	2.02	2.738 (2)	143

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

Fig. 1

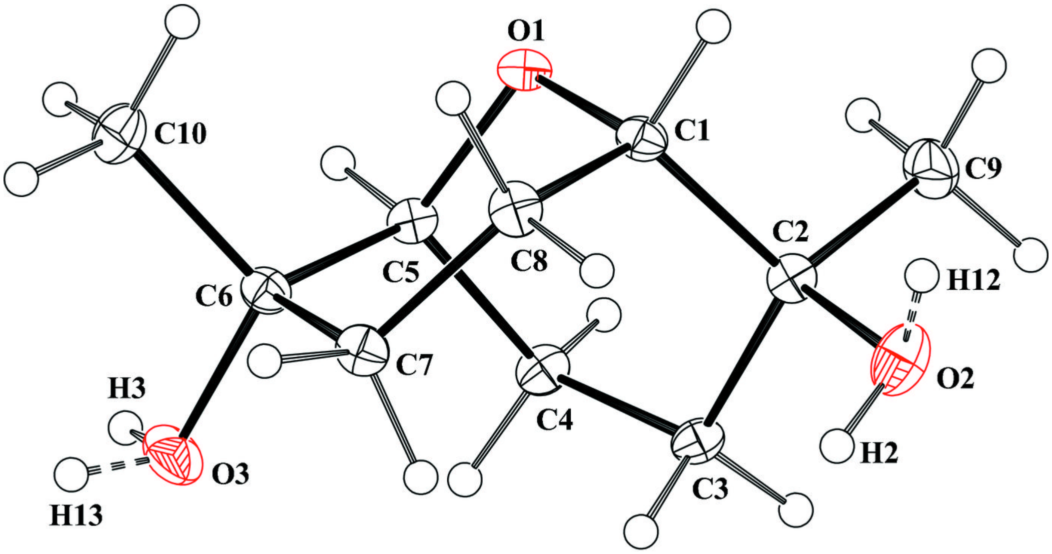

Fig. 2

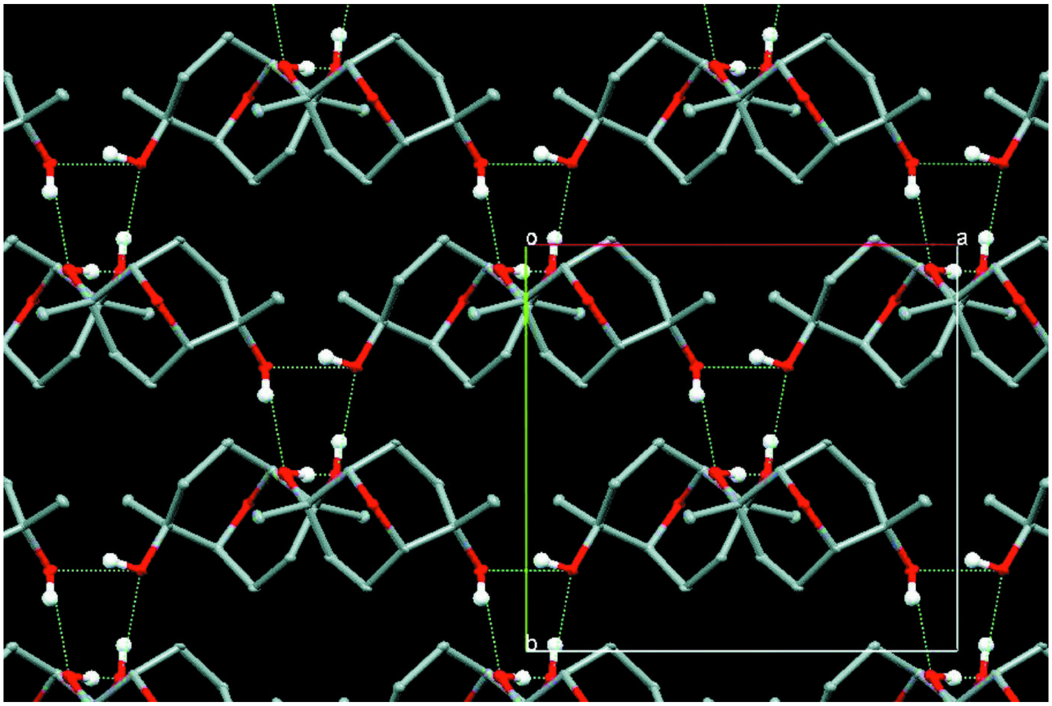

Fig. 3