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1-Cyclohexylmethoxymethyl-5-[2-hydroxy-1-(hydroxymethyl)ethylamino]cyclohexane-1,2,3,4-tetraol

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.036
wR factor = 0.087
Data-to-parameter ratio = 9.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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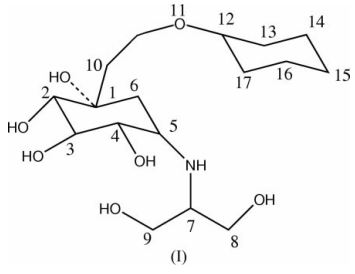
The title compound, $C_{17}H_{33}NO_7$, contains two six-membered rings which adopt chair conformations. The molecular packing is governed by intermolecular O—H···O hydrogen bonding.

Comment

As an antidiabetic medicine, voglibose {5-[2-hydroxy-1-(hydroxymethyl)ethylamino]-1-(hydroxymethyl)cyclohexane-1,2,3,4-tetraol} shows excellent inhibitory activity against α-glucosidase and is therefore useful for hyperglycemic symptoms and various disorders caused by hyperglycemia (Satoshi *et al.*, 1986; Vichayanrat *et al.*, 2002).



(I)

The molecule of the title compound, (I) (Fig. 1), contains two non-aromatic six-membered rings. Both rings, *A* (C1–C6) and *B* (C12–C18), adopt chair conformations. These two rings are roughly in the same plane, with atoms O1 and N on the same side of this plane. One of the rings (*B*) is disordered, with three of the C atoms distributed on two sites with approximately equal occupancy. In addition, one of the two hydroxymethyl groups attached to N is disordered over two positions.

The molecular packing of (I) exhibits intermolecular O—H···O hydrogen bonding (Table 2 and Fig. 2).

Experimental

The mixture which was obtained in the synthesis of voglibose containing the title compound was collected and separated by silica-gel column chromatography using increasing proportions of acetone in methanol (starting at 10%) as eluants. The crude title compound (55 mg) was obtained. After repeated purification by column chromatography on Sephadex LH-20 eluting with H_2O–methanol (3:1), 25 mg of the pure title compound (m.p. 455–456 K) was obtained. The compound was identified from ^{13}C NMR spectra (p.p.m.): C1 (99.89), C2 (53.96), C3 (26.10), C4 (30.84), C5 (75.40), C6 (79.08), C7 (58.67), C8 (76.12), C9 (76.95), C10 (56.31), C11 (70.82), C12 (61.34), C13 (36.50), C14 (173.40), C15 (45.82), C16 (56.23), C17 (19.56). Crystals suitable for X-ray analysis were obtained by slow evaporation of an aqueous methanol solution (1:1, 5 ml) at room temperature.

Crystal data

$C_{17}H_{33}NO_7$
M_r = 363.44
Monoclinic, $P2_1$
a = 8.437 (2) Å
b = 6.853 (1) Å
c = 16.011 (3) Å
β = 94.88 (2)°
V = 922.4 (3) Å^3
Z = 2
D_x = 1.309 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 33 reflections
θ = 4.7–15.2°
μ = 0.10 mm^{-1}
T = 296 (2) K
Chunk, colorless
0.56 × 0.50 × 0.20 mm

Data collection

Siemens $P4$ diffractometer
ω scans
Absorption correction: none
4872 measured reflections
2590 independent reflections
2131 reflections with $I > 2\sigma(I)$
R_{int} = 0.011
θ_{max} = 28.7°
$h = -10 \rightarrow 11$
$k = -8 \rightarrow 9$
$l = -21 \rightarrow 21$
3 standard reflections
every 97 reflections
intensity decay: 2.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.088
S = 0.97
2590 reflections
285 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0556P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.4364 (19)	O5—C11	1.424 (3)
O2—C2	1.431 (2)	O6—C8	1.406 (3)
O3—C3	1.430 (2)	N—C7	1.467 (2)
O4—C4	1.429 (2)	N—C5	1.480 (2)
O5—C10	1.417 (2)		
C10—O5—C11	109.46 (14)	C10—C1—C2	106.73 (13)
C7—N—C5	115.07 (13)	C6—C1—C2	109.03 (14)
O1—C1—C10	108.79 (15)	N—C5—C4	108.93 (12)
O1—C1—C6	110.94 (14)	N—C5—C6	110.85 (15)
C10—C1—C6	112.14 (13)	C4—C5—C6	110.15 (14)
O1—C1—C2	109.10 (13)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N	0.82	2.03	2.763 (2)	148
O2—H2···O3^{i}	0.82	1.91	2.7264 (19)	171
O3—H3···O6^{ii}	0.82	1.95	2.748 (2)	166
O3—H3···O7^{iii}	0.82	2.66	3.067 (3)	112
O4—H4···O2^{iv}	0.82	2.06	2.808 (2)	152
O6—H6···O1^{iv}	0.82	1.92	2.729 (2)	171
O6—H6*D*···O7*B*	0.85	2.52	2.969 (6)	114
N—H1N···O4	0.83 (2)	2.32 (2)	2.803 (2)	117.3 (16)
O7—H7···O4^{ii}	0.82	2.31	3.074 (3)	156
O7*B*—H7*B*···O2^{iii}	0.82	2.15	2.877 (5)	147
O7*B*—H7*B*···O3^{iii}	0.82	2.52	3.008 (7)	120

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

One of the rings (*B*) was disordered with three of the C atoms distributed over two sites with a distribition ratio of approximately 50:50. The H atom on the N atom was refined freely. The hydroxy H atoms were located in difference Fourier syntheses but were treated as riding, together with all the other H atoms, with O—H = 0.82 and C—H = 0.97–0.98 Å and U_{iso} = 1.2U_{eq} or 1.5U_{eq} of the carrier atom. Friedel reflections were merged before the final refinement. The disordered parts of the molecule were constrained to chemically reasonable geometry with the help of available tools in *SHELXL*97 (Sheldrick, 1997).

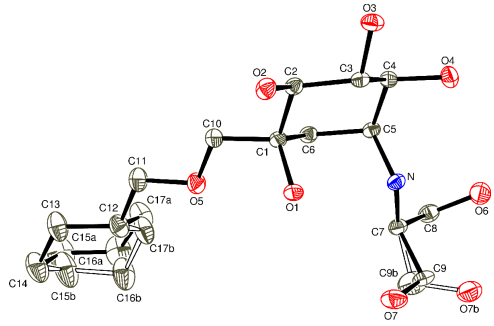


Figure 1
View of the molecule of the title compound, showing 30% probability displacement ellipsoids. Hydrogen atoms have been omitted, and all disorder components are shown.

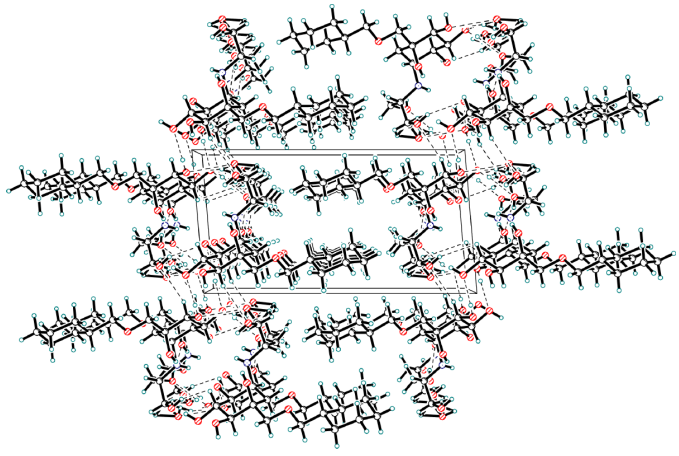


Figure 2
The hydrogen bonding in (I), viewed normal to the (001) plane. Hydrogen bonds are shown as dashed lines.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Siemens, 1991); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* and *ORTEP*III (Burnett & Johnson, 1996) and *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL*.

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